Microstructure and properties of semi-solid injection molded magnesium-based materials

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Abstract: The preparation of semi-solid slurry is the most important link in the semi-solid forming process. The high viscosity of the semi-solid metal slurry contributes to the uniform dispersion of GNPs in the magnesium matrix. Systematic research on the influence of stirring rate on semi-solid slurry is of great significance for the homogenized preparation of magnesium-based composites. Graphene nanosheet reinforced magnesium matrix composites at different stirring rates were prepared by semi-solid injection molding process.It was observed in the semi-solid microstructure that both excessively high (170 r/min) and excessively low (110 r/min) stirring rates would lead to the formation of residual solid phase clusters, and at the same time, a certain degree of agglomeration phenomenon was found in GNPs. During the slurry preparation process, an increase in the stirring rate will lead to an increase in the feeding rate, resulting in insufficient stirring time. Therefore, only by balancing the stirring speed and time can the optimal preparation of semi-solid mixed slurry be achieved. It was found that when the stirring rate reached 150r/min, GNPs are uniformly distributed in the magnesium matrix, The performance of composite materials is the most balanced, The tensile strength reaches 228.6MPa and the Vickers hardness is 92.7HV.

Keywords: Semi-solid injection molding; Semi-solid slurry; Stirring rate; Stirring time; Residual solid phase.

1 Introduction

Magnesium alloys, as the lightest metal structural materials, have attracted much attention in lightweight fields such as automobiles, aerospace, and electronic devices due to their excellent specific strength, damping performance, and recyclability[1-3]. However, Traditional magnesium alloy forming processes, including die casting and extrusion casting, exhibit notable limitations that hinder their industrial application. Firstly, the high reactivity of magnesium melt at elevated temperatures induces severe oxidation and combustion risks, compromising process stability. Secondly, rapid solidification characteristics during these processes often generate internal defects such as gas pores and shrinkage cavities, which detrimentally affect the mechanical performance of final products. Furthermore, when manufacturing geometrically complex components, these methods frequently encounter technical challenges including incomplete mold filling and thermal stress-induced cracking, particularly in thin-walled or intricately contoured structures. While traditional alloying strategies can enhance the absolute strength of magnesium alloys, such improvements remain insufficient for demanding operational scenarios-particularly in critical applications requiring thin-walled load-bearing components or high-stress structural parts. This performance gap underscores the pressing need for synergistic innovations in advanced material architectures (such as nano-reinforced composites) and novel processing techniques (such as additive manufacturing or severe plastic deformation) to achieve revolutionary strength-to-weight ratio enhancements.

Semi-solid injection molding technology (SSM) provides new ideas for the above-mentioned problems. This technology maintains the alloy melt in a solid-liquid coexistence regime (solid fraction: 30-60%), thereby integrating the high fluidity of liquid-state forming with the low shrinkage attributes inherent to solid-state processes. This process effectively minimizes defect formation, refines grain structures, and enhances dimensional precision through controlled solidification dynamics[4,5]. Research demonstrates that semi-solid magnesium alloys exhibit 20-30% enhancement in mechanical properties compared to traditional die castings, primarily attributed to their characteristic spherical grain structure with uniform distribution. Furthermore, the forming temperature is reduced by 100-150℃ relative to conventional processes. This temperature reduction decreases oxidation kinetics by 40-60%, while simultaneously extending mold service life through mitigated thermal fatigue damage during cyclic production.

Improving the strength-modulus of magnesium alloys merely through alloying still limits their application potential in the industrial field. To further expand the application boundaries of magnesium alloys, Researchers have focused on metal matrix composites (MMCs) by incorporating reinforcement agents, including conventional ceramic particles (SiC, Al_2O_3), carbon fibers, and advanced nano-reinforcements such as graphene or carbon nanotubes[6-8]. The advancement of magnesium matrix composites drives technological innovation in lightweight engineering, particularly through enhanced specific strength and vibration damping capabilities. These material developments enable breakthrough applications in transportation sectors, including new energy vehicle battery enclosures (achieving 30-45% weight reduction) and aerospace structural components with optimized payload capacities, It is of strategic significance for achieving the innovation of structure-function integrated materials under the "dual carbon" goals[9].

Since its discovery, Graphene has been regarded as an ideal reinforcing material for traditional metals, especially lightweight magnesium alloys, due to its excellent mechanical strength, thermal conductivity and electronic effects[10]. Graphene Nanoplates (GNPs), as derivatives of graphene, maintain the excellent properties of graphene while having a relatively low production cost. They are highly promising nano-carbon material reinforcements to be developed as composite materials. However, due to the large specific surface area and strong interlayer van der Waals forces of graphene, adding it to the matrix through traditional composite material preparation methods (such as stirred casting [11], powder metallurgy [12]) will face uneven distribution and severe agglomeration of the reinforcing phase, poor interfacial reactions, and high process energy consumption, thereby reducing the reinforcing effect. Semi-solid injection technology has the following unique advantages in solving these problems: The high viscosity of semi-solid slurries can inhibit the sedimentation of enhanced phases and the tendency of flow agglomeration, Uniform distribution is achieved by combining the effect of the shear flow field; A lower molding temperature reduces the harmful reactions between the matrix and the reinforcing material, Meanwhile, the thixotropy of the semi-solid slurry is conducive to the formation of mechanical interlocking interfaces; It is suitable for the integrated molding of complex structural components, avoiding the damage to the reinforcing phase caused by subsequent machining[13-15]。

At present, a certain foundation has been established for the research of semi-solid injection-magnesium-based composites. However, the microstructure evolution mechanism, the co-deformation behavior of the reinforcing phase/matrix, and the process-performance correlation law still need to be further explored. The preparation quality of semi-solid slurry is an important influencing factor of the forming quality of castings. Chen et al. [16] from North University of China investigated the influence of screw stirring rate and injection speed on the microstructure, dispersion of graphene nanoplatelets (GNPs), and mechanical properties of AZ91D-GNPs composites.It has been observed that excessively high or low stirring rates can result in an uneven distribution of GNPs within the semi-solid slurry. Additionally, the high-speed filling process may exert a significant stirring effect on the reinforcement. Gu et al. [17], researchers from Shanghai Jiao Tong University, fabricated SiC-Al_2O_3 hybrid-reinforced magnesium matrix composites via a semi-solid injection molding process and discovered that the incorporation of the reinforcement significantly decreased the residual solid phase content in the semi-solid melt. In the semi-solid injection molding process, the screw stirring rate has an important influence on the quality of the mixed slurry. It is essential not only to ensure that the dendrites are fragmented into fine and uniformly spherical particles but also to guarantee that the mixed slurry exhibits excellent fluidity. Additionally, through screw stirring, the reinforcement should be dispersed as evenly as possible within the mixed slurry[18,19]. Therefore, a systematic investigation into the influence of screw stirring rate on the microstructure morphology evolution of semi-solid slurry and its regulation of the performance of the final casting constitutes an effective approach to overcoming the technical bottlenecks associated with high-performance magnesium-based materials.

In this study, GNPs/AZ91D composites were fabricated at varying stirring rates using the semi-solid injection molding process， The study investigated the influence of stirring rate on the evolution of the semi-solid microstructure in magnesium-based materials, as well as the interfacial bonding mechanisms between graphene and the magnesium matrix, The study analyzed the influence mechanism by which the interface between graphene and the matrix, in synergy with the semi-solid microstructure, affects the mechanical properties of composite materials.

2 Experimental section

2.1 Raw material

The reinforcing material is GNPs (purity 99.5%), with a grayish-black powder morphology, a diameter of 5-10 μm, and a thickness of 3-10 nm. In the semi-solid injection molding process, AZ91D particles machined to a size of 3-5mm are used as the matrix raw material. The chemical composition of AZ91D magnesium alloy particles is shown in Table 1.

Table 1. The chemical composition of AZ91D alloy (mass fraction, wt.%)

Al	Zn	Mn	Si	Fe	Mg
8.30	0.54	0.23	≤0.05	≤0.04	Bal.

2.2 Preparation of AZ91D-GNPs composites

Figure 1 presents a schematic illustration of the semi-solid injection molding process. An V-type powder mixer was employed to thoroughly blend AZ91D granules with a Graphene Nanoplatelets (GNPs) content of 0.6 wt. %. The mixer operates at a rotational speed of 20 revolutions per minute for a duration of 30 minutes to achieve a homogeneous mixture of GNPs and AZ91D particles. Subsequently, the mixture is transferred into the preheated barrel of the magnesium alloy injection molding machine. As it moves from the feed port to the nozzle, the preheating temperature progressively increases from 300℃ to 600℃. The screw rotates at speeds of 110, 130, 150, or 170 revolutions per minute to stir and mix the particles, thereby forming a semi-solid slurry, the material is injected into the mold, which has been preheated to 275℃, at an injection speed of 1.5 meters per second. Thus, GNPS-reinforced magnesium matrix composites were successfully fabricated by stirring at varying rotational speeds.

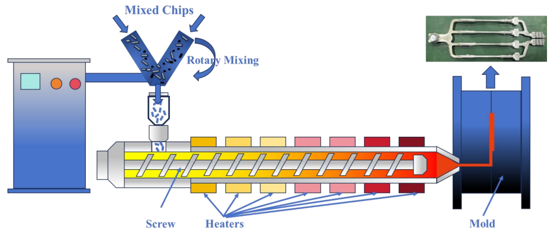

Fig. 1: Schematic diagram of semi-solid injection molding process

2.3 Microstructure characterization and mechanical property testing

The microscopic morphology of the specimens was systematically investigated using optical microscopy (OM, Leica DMi8), scanning electron microscopy (SEM, HITACHI SU5000), high-resolution transmission electron microscopy (HR-TEM), and energy dispersive spectroscopy (EDS, JEOL JEM-F200). The tensile mechanical properties of the specimens were evaluated using an electronic universal testing machine (SHIMADZU AG-X plus). Five tests were performed on each sample to evaluate its mechanical properties. Tensile tests were performed using specimens with a diameter of 5 mm and a gauge length of 25 mm, in accordance with standard testing protocols. The Vickers hardness of the specimens was measured using the TMHVS-1000 fully automatic digital microhardness tester, with a load of 500 gf and a dwell time of 15 seconds.

3 Results and discussion

3.1 Microstructure Evolution

The optical micrographs of the microstructure morphology for 0.6 wt.% GNPs/AZ91D magnesium-based composites at various screw stirring rates (110, 130, 150, and 170 r/min) are presented in Figure 2. A typical semi-solid structure consisting of a spherical residual solid phase and a primary α-Mg solid phase can be observed. Furthermore, as the stirring rate increases, the content of the residual solid phase rises while its size diminishes. When the stirring rate is 110 r/min, the low shear strength results in the formation of noticeable clusters of solid particles within the mixture. With the increase in stirring rate, the residual solid phase becomes gradually and more uniformly dispersed. When the stirring rate reaches 170 r/min, there is a significant increase in the number of residual solid-phase particles. It has been demonstrated that increasing the stirring rate can effectively shear and disperse residual solid particles. However, as the stirring rate increases, the heating time for the mixed particles is reduced, leading to an increase in the solid content of the semi-solid melt. The grain size analysis conducted using Image Pro software demonstrated that as the stirring rate increased from 110 to 150 r/min, the average grain size decreased from 21.5 μm to 13.3 μm, indicating a significant refinement of the grains. Meanwhile, the shape factor increased from 0.61 to 0.83, suggesting a transformation of the primary α-Mg grains from dendritic structures to nearly spherical morphologies. When the stirring rate was further increased to 170 r/min, the grain size exhibited a trend of coarsening. The increase in the residual solid phase and the coarsening behavior of grains are attributed to the reduced residence time of the AZ91D and GNPs mixed particles within the heated barrel[20]. The conveying speed increases with the rise in stirring speed, leading to insufficient heating time for the mixed particles within the barrel. Additionally, the solid-phase fraction of the semi-solid slurry is relatively high, causing it to enter the nozzle before complete melting and resulting in the coarsening of unmelted solid-phase grains.

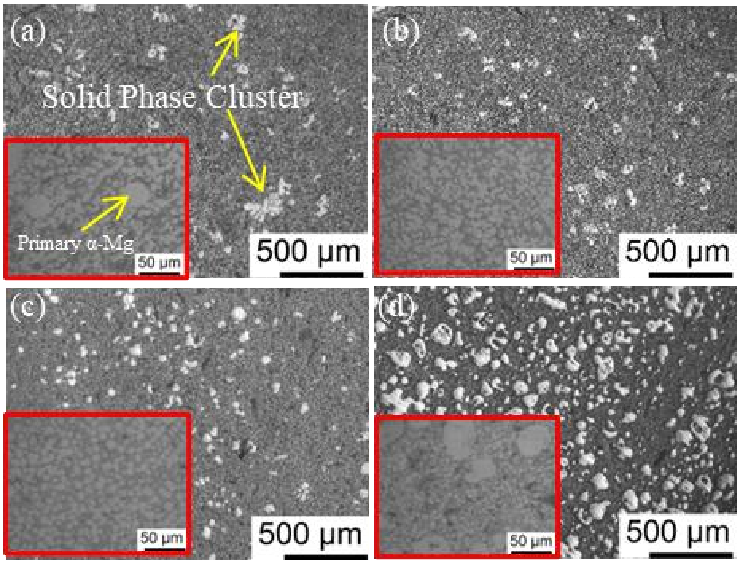

Fig. 2: Microscopic morphology of composites at different stirring rates

3.2 Distribution of GNPs

As illustrated in Figure 3, the backscattering SEM images of GNPs/AZ91D magnesium-based composites at varying stirring rates are presented, The presence of black strips in the SEM image, which are the added graphene nanosheets, can be confirmed through EDS point scanning and surface scanning, It is evident that the graphene nanoplanes (GNPs) are closely integrated with the surrounding magnesium matrix without any noticeable defects, which confirms the successful incorporation of GNPs into the matrix. Furthermore, the distribution of GNPs under different stirring rates is systematically assessed based on their size and distribution density. It can be observed that at a relatively low stirring rate of 110 r/min, significant agglomeration of graphene nanoparticles (GNPs) occurs. As depicted in Figures 3(b) and (c), an increase in the stirring rate (130 and 150 r/min) leads to a notable reduction in agglomeration, resulting in a more uniform size and distribution of GNPs. As depicted in Figure 3(d), upon further increasing the stirring rate to 170 r/min, it is evident that the agglomeration of graphene nanoparticles (GNPs) intensifies significantly once again, This is because an increase in the stirring rate enhances feeding efficiency while reducing screw stirring time, thereby enabling the mixed particles to be transported to the head of the material pipe within a shorter period. However, the high concentration of residual solid particles and insufficient screw stirring weaken the dispersion effect of GNPs.

In conclusion, the stirring rate should be maintained within an optimal range to ensure both sufficient stirring and shear strength for dispersing GNPs in the matrix while minimizing adverse effects caused by increased conveying efficiency and reduced screw mixing time on GNP dispersion.

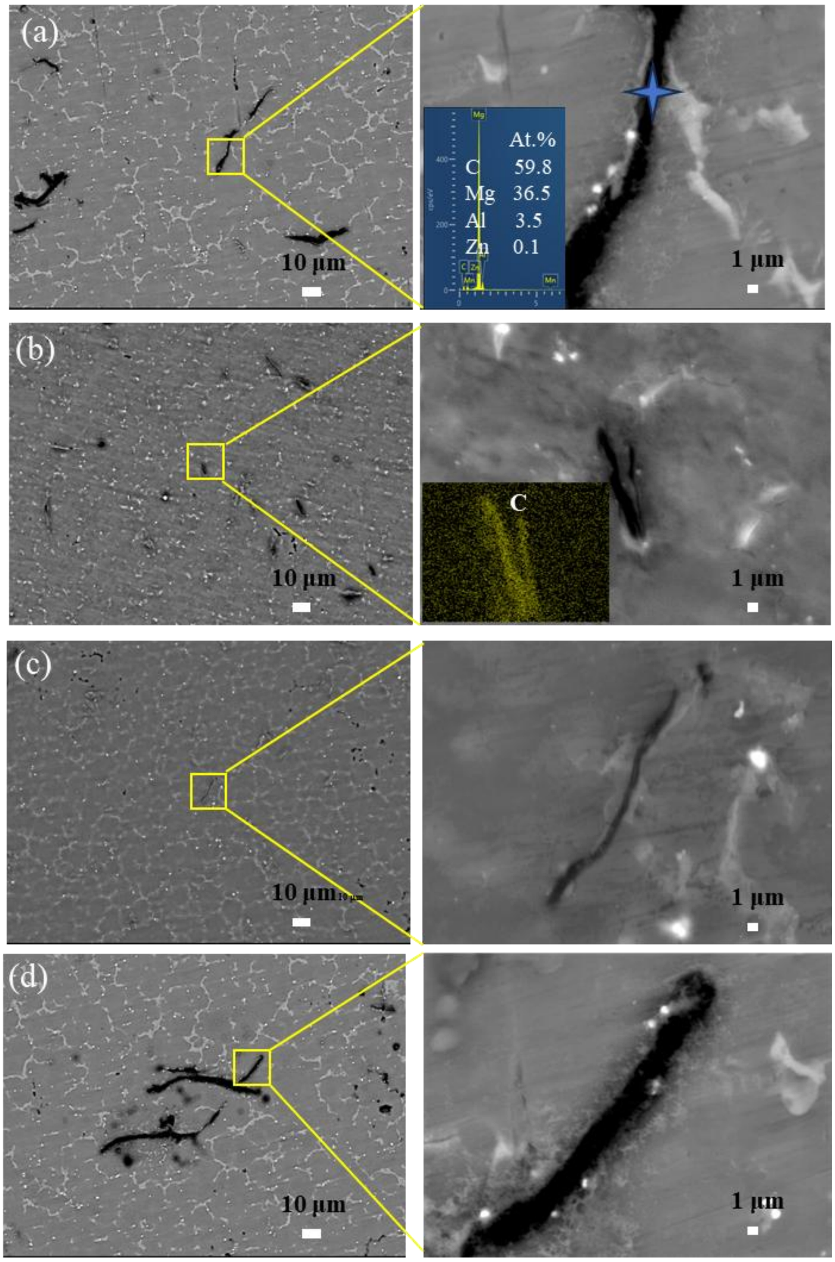

Fig. 3: Dispersion of GNPs in the Mg matrix at different stirring rates

3.3 Mechanical properties

The hardness and tensile properties of GNPs/AZ91D magnesium matrix composites at various stirring rates are presented in Figure 4. It can be observed that the hardness and tensile strength exhibit a trend of initially increasing and subsequently decreasing. When the stirring rate was increased from 110 r/min to 150 r/min, the hardness of the as-cast sample reached a peak value of 92.7 HV, while the tensile strength attained 228.6 MPa. However, when the stirring rate is progressively increased to 170 r/min, the excessive stirring rate combined with insufficient stirring duration results in a high solid-phase melt, which in turn causes agglomeration of graphene nanoplates (GNPs) within the matrix. Graphite layers formed through agglomeration can be observed within the matrix, suggesting that graphene nanoplatelets (GNPs) lose their single-layer graphene characteristics and transition into graphite. This transformation results in a reduction of the enhancement efficiency. The mechanical properties of the sample with a stirring rate of 170 r/min are measured at only 177.5 MPa, which is even lower than those of the sample with a stirring rate of 110 r/min. In conclusion, when the stirring rate is set to 150 r/min, the GNPs/AZ91D composite material exhibits optimal mechanical properties.

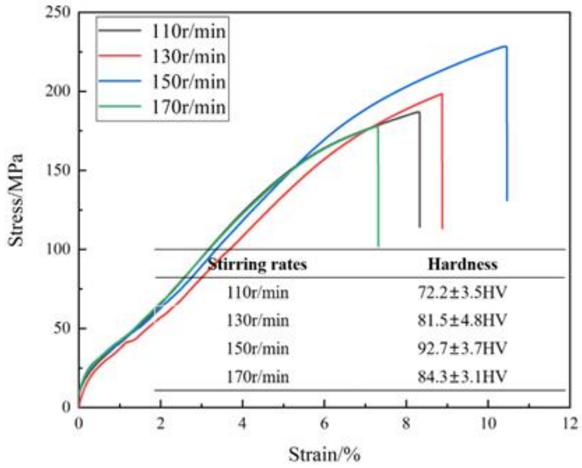

Fig. 4: Mechanical properties of composites at different stirring rates

3.4 Fracture mechanism of composite materials

The incorporation of GNPs altered the stress state of the composite material under loading conditions. This modification subsequently influenced its fracture mechanism, transitioning the fracture mode from purely brittle fracture to a hybrid fracture characterized by both toughness and brittleness[21]. Figure 5 presents the SEM images of the representative tensile fracture surface of GNPs/AZ91D composites, and the presence of GNPs is confirmed and validated through EDS point scanning and surface scanning analysis. It can be observed that the typical fracture morphologies of GNPs/AZ91D composites primarily consist of two forms: GNPs fracture and GNPs pull-out. In Figure 5(a), the edges of the graphene nanoplatelets (GNPs) appear smooth and flat, indicating a clean pull-out from the substrate. The relatively low interfacial bonding strength leads to direct detachment from the substrate under applied loading conditions. In contrast, as shown in Figure 5(b), the edges of the GNPs exhibit irregular morphology. This suggests that the GNPs have fractured due to the higher interfacial bonding strength, which enables effective stress transfer from the substrate to the GNPs. Under such conditions, the transferred load exceeds the maximum tensile capacity of the GNPs, resulting in fracture.

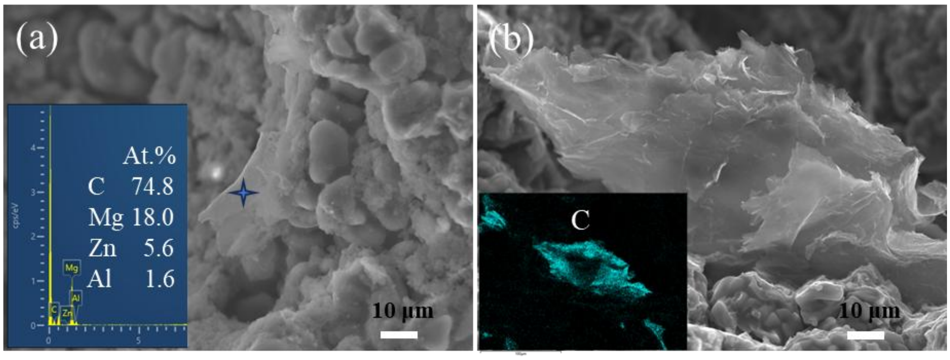

Fig. 5: Fracture morphology and fracture mode of magnesium matrix composites

3.5 Influence of the screw mixing process on the dispersion mechanism of GNPs in the matrix

The screw stirring process plays a crucial role in achieving the uniform dispersion of GNPs within the AZ91D magnesium alloy matrix. Figure 6 presents a schematic illustration of the dispersion process of graphene nanoplatelets (GNPs) during the screw stirring operation, During the semi-solid injection molding process, the mixture of graphene nanoplatelets (GNPs) and AZ91D magnesium alloy experiences four distinct stages within the barrel: shear mixing, formation of the primary molten pool, expansion of the molten pool, and semi-solid homogenization. This ultimately results in the production of a semi-solid mixed slurry.[22,23]。

When the mixed particles enter the material pipe, they are subjected to the compressive and shearing forces generated by the screw rotation. The alloy particles experience collision and friction against each other, leading to cracking, fragmentation, and compaction of the particles. Subsequently, GNPs and AZ91D particles establish intimate contact and infiltrate into the particle cracks. With the continuous operation of the heater, the temperature of the material tube gradually increases in the forward direction along the flow path of AZ91D particles. This temperature rise is attributed to the synergistic effects of material tube heating and screw shear-induced stirring. The region of severe plastic deformation on the surface layer of AZ91D alloy particles will initially melt, leading to the formation of a primary molten pool. As the screw rotates, it generates compressive forces and shear stresses, causing the material within the barrel to experience a continuous rise in temperature.

Consequently, the solid boundary surrounding the melt pool progressively melts, leading to the expansion and growth of the melt pool. Meanwhile, relatively proximal small melt pools will coalesce to form larger melt pools. As the solid phase continues to melt, these larger melt pools will progressively expand, interconnect with other melt pools within the alloy particles, and ultimately integrate into a cohesive alloy melt. At this stage, graphene nanoplatelets (GNPs) will be thoroughly mixed into the molten pool to ensure uniform dispersion. With the ongoing advancement of shearing and heating processes, the particle morphology of the solid phase progressively evolves toward refinement and rounding. Owing to the limited screw stirring time (30-40 seconds) and the relatively low melt temperature (590-600° C), it is not feasible to completely melt all AZ91D particles, resulting in some residual spherical solid phases remaining within the alloy melt. Stirring the material in a semi-solid state can effectively diminish the buoyancy of graphene nanoplatelets (GNPs), thereby facilitating their stabilization within the semi-solid alloy melt. With the effect of shear heating, the mixed particles transition into a solid-liquid mixed phase at the tail end of the material tube. The remaining solid particles undergo spheroidization, while the graphene nanoplates (GNPs) achieve further dispersion within the melt. This process ultimately results in the formation of a uniformly distributed semi-solid mixed slurry.

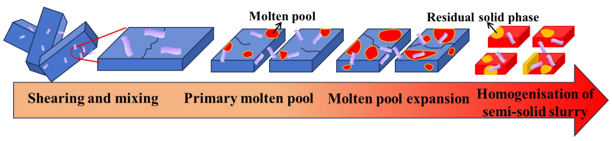

Fig. 6: Evolution of GNPs-AZ91D mixed particles during screw mixing

4 Conclusions

The impact of stirring rate on the mechanical properties of GNPs/AZ91D magnesium matrix composites was examined through microstructure characterization and mechanical property testing. The key findings of this study can be summarized as follows:

（1） Magnesium-based composites were fabricated via semi-solid injection molding using graphene nanoplatelets (GNPs) powder and AZ91D alloy particles as raw materials. Microstructural characterization and analysis demonstrated that at a stirring rate of 150 r/min, the residual solid content and dispersion of GNPs were optimized, resulting in a tensile strength of 228.6 MPa and a hardness of 92.7 HV.

（2） With the increase in stirring rate (from 110 r/min to 150 r/min), the grain structure was significantly refined, the fraction of residual solid phase gradually increased, and the primary α-Mg grains transitioned from dendritic to nearly spherical morphology. However, with a further increase in stirring rate (from 150 r/min to 170 r/min), the effective stirring time was substantially reduced, leading to a decrease in both the residual solid phase fraction and the uniformity of the reinforcing material distribution.

（3） The incorporation of graphene nanoplatelets (GNPs) in magnesium-based composites alters the fracture mechanism from purely brittle to a mixed mode of brittle and ductile fracture. This transformation primarily facilitates load transfer within the composite structure. Two distinct modes of failure, namely fracture and pullout, are observed on the fracture surface. The presence of fracture indicates a strong bonding strength between GNPs and the matrix, which significantly enhances the mechanical properties of the composite material.

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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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