Microstructure and Properties of AZ91D Magnesium Alloy Processed by Forced Convection Stirring Rheo-Die-Casting (Postprint)

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Abstract

Based on the principle of forced convection mixing, a Forced Convection Mixing (FCM) slurry preparation device was successfully developed independently and integrated with a die casting machine. Using AZ91D magnesium alloy tensile test specimens as an example, an integrated rheo-die casting process encompassing slurry preparation, transfer, and forming was achieved. The evolution of microstructural characteristics in rheo-die castings under different FCM process parameters was investigated, the differences in mechanical properties of die castings produced by different processes were compared, and the microstructure formation mechanism and solidification behavior of the FCM rheo-die casting process were analyzed. The results indicate that FCM process parameters exert a significant influence on the microstructure of castings; appropriately increasing the screw speed or decreasing the barrel temperature is conducive to improving the microstructural morphology of the formed components. The FCM rheo-die casting process can not only produce formed components with fine, round, and uniformly distributed internal microstructures, but also significantly enhance the mechanical properties of the formed components. Compared with conventional die castings, the yield strength of FCM rheo-die castings shows minimal variation, while the tensile strength and elongation are increased by 12.5% and 80.0%, respectively. Compared with castings subjected to T4 and T6 heat treatments, the as-cast tensile specimens exhibit the lowest tensile strength, with yield strength and elongation falling between those of T4 and T6 treatments.

Full Text

Preamble

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Abstract

Based on the forced convection mixing (FCM) principle, a self-developed FCM semisolid slurry preparation device was successfully developed. Taking AZ91D magnesium alloy tensile parts as an example, the rheo-diecasting process that integrates slurry preparation, transportation, and forming was achieved by combining the device with a diecasting machine. Microstructural characteristics of FCM rheo-diecasting parts under different processing parameters were investigated, mechanical properties of AZ91D alloy parts produced by different processes were compared, and the formation mechanism and solidification behavior of semisolid slurry in the FCM rheo-diecasting process were analyzed. The results show that processing parameters have a significant effect on the microstructures of parts; appropriately increasing rotation speed or decreasing barrel temperature is beneficial for optimizing the microstructure. The process can not only produce parts with fine, spherical, and uniformly distributed primary α-Mg particles, but also significantly improve the mechanical performance of parts. Compared with traditional diecasting, the yield strength remains essentially unchanged, while the ultimate tensile strength and elongation are increased by 12.5% and 80.0%, respectively. Furthermore, compared with parts subjected to T4 and T6 heat treatments, the as-cast tensile parts exhibit the lowest ultimate tensile strength, with yield strength and elongation falling between those of T4 and T6 treated parts.

KEY WORDS AZ91D magnesium alloy, forced convection mixing (FCM), rheo-diecasting, microstructure evolution, mechanical properties, heat treatment

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Magnesium alloys are currently the lightest metallic materials in engineering applications, offering a unique combination of high specific strength and stiffness, good damping capacity, excellent machinability, and easy recyclability. These properties have led to their recognition as a "green engineering material for the 21st century" with significant potential for automotive and 3C (computer, communication, and consumer electronics) industries. Common magnesium alloy forming methods include diecasting, semisolid casting, and squeeze casting, among which diecasting is the most widely adopted, with the majority of practical magnesium alloy components being diecast parts. While magnesium alloy diecast products offer advantages such as good dimensional stability, high productivity, and long die life, they also suffer from serious limitations that restrict broader application, including high levels of inclusions and porosity, difficulty in heat treatment after forming, low mechanical properties, and poor near-net-shape capability.

To improve internal defects and enhance mechanical properties of diecast parts, several technologies have been developed in recent years, including vacuum diecasting, oxygen-filled diecasting, and semisolid rheo-diecasting. Among these, semisolid rheo-diecasting has received widespread attention both domestically and internationally due to its broad application range, short process flow, low forming temperature, dense casting structure, and potential for heat treatment strengthening, making it a hot topic in semisolid processing. The preparation of semisolid slurry is a critically important step in rheoforming technology development. However, to date, the continuous and rapid preparation of semisolid slurry remains a bottleneck restricting the rapid development and large-scale industrial application of this technology.

In this work, widely used AZ91D magnesium alloy was selected as the experimental material, and a self-designed mechanical property test die was used as the experimental diecasting mold. A self-developed mechanical stirring device—the forced convection mixing (FCM) machine—was employed to prepare semisolid slurry, which was combined with a diecasting machine to achieve an integrated rheo-diecasting process encompassing slurry preparation, transportation, and forming. Based on this setup, the influence of different FCM process parameters on the microstructure of rheo-diecast tensile parts was systematically investigated. The mechanical properties of tensile parts produced by traditional diecasting, rheo-diecasting, and different heat treatment regimes were compared, and the microstructure formation mechanism and solidification behavior of the FCM rheo-diecasting process were analyzed.

1.1 Experimental Equipment

[Figure 1: see original paper] shows the structural diagram of the laboratory-developed FCM device. The equipment consists primarily of a drive unit, shearing/stirring device, temperature control system, and discharge mechanism. A stepless speed-regulating motor drives the stirring shaft through transmission gears. The shearing/stirring mechanism comprises a stirring chamber and stirring shaft, with the latter equipped with spiral ribbons suitable for stirring viscous fluids. The rotation speed can be adjusted to generate fluid convection of the alloy melt within the stirring chamber. To prevent melt solidification and adhesion on the chamber walls, a graphite liner is installed on the inner wall of the stirring chamber. Heating and cooling elements on the outer wall of the barrel enable precise temperature control via a temperature control box. The discharge mechanism consists of a discharge handle, core bar, and blockage. After slurry preparation, rotating the discharge handle lifts the blockage, allowing the prepared semisolid slurry to flow out through a preheated collection crucible for rapid transfer to the shot sleeve of a DC350J cold-chamber diecasting machine for rheo-diecasting. The FCM rheo-diecasting process is schematically illustrated in [Figure 1: see original paper].

The experimental material was commercial high-purity AZ91D magnesium alloy ingot with a chemical composition (mass fraction, %) of: Al 9.03, Zn 0.75, Mn 0.145, Si 0.033, and Mg balance. Differential scanning calorimetry (DSC) analysis determined its liquidus and solidus temperatures to be 595 °C and 470 °C, respectively.

AZ91D magnesium alloy ingots were preheated in a melting furnace at 200 °C, then heated to 680 °C for melting. To prevent oxidation and combustion of the magnesium alloy melt, RJ-2 flux was used as protective cover during melting. The melt was scooped out and cooled to 640 °C before being poured into the FCM device with preset process parameters. Within the barrel at a temperature below the alloy' s liquidus, the alloy melt generated convection under the rotation of the stirring shaft. Through the combined action of cooling and convection, semisolid slurry was produced. The process parameters were: screw rotation speed 100–700 r/min, barrel temperature 560–580 °C, pouring temperature 640 °C, and stirring time 25 s. Subsequently, the discharge handle was rotated to release the slurry, and the collected slurry was rapidly transferred to the diecasting machine shot sleeve for casting. For comparison, traditional diecast tensile parts in this work were produced at a pouring temperature of 640 °C, with diecasting parameters identical to those used for FCM rheo-diecasting: casting pressure 80 MPa, slow shot speed 0.3 m/s, fast shot speed 1.2 m/s, and mold temperature 220 °C.

The schematic diagram of the rheo-diecast tensile test sample is shown in [Figure 2: see original paper]. Samples were cut from the same location of tensile parts, then ground and polished. After etching with 4% nitric acid (volume fraction) in alcohol, rinsing, and drying, microstructural observation and analysis were conducted using a NEOPHOT 21 optical microscope (OM) and Cambridge S-360 scanning electron microscope (SEM). Image Tool software was used to analyze the size and shape factor of primary solid phase particles. The primary solid phase size was represented by equivalent circle diameter D, and the shape factor was represented by F, where a larger F value closer to 1 indicates rounder primary phase particles. The specific formulas are:

$$D = 2\sqrt{A/\pi}$$

$$F = 4\pi A/P^2$$

where A is the grain area and P is the grain perimeter. Tensile tests were performed on an MTS810 universal testing machine according to GB/T 228-2002 at a strain rate of 1 mm/min, with mechanical property values representing the average of five test specimens.

The heat treatment regimes employed were solution treatment (T4) and solution treatment plus artificial aging (T6). The solution treatment process involved heating at 10 °C/min to 260 °C, holding for 1 h, loading the tensile parts (covered with graphite powder under Ar atmosphere), then heating at 1 °C/min to 415 °C, holding for 12 h, followed by furnace removal and air cooling. The aging process involved heating at 6 °C/min to 175 °C, holding for 24 h, then air cooling.

2.1 Effect of Screw Rotation Speed on Rheo-Diecast Microstructure

[Figure 3: see original paper] presents optical micrographs of FCM rheo-diecast tensile parts produced at a barrel temperature of 560 °C and screw rotation speeds of 100–700 r/min. Compared with the coarse dendritic structure of traditional diecast parts, FCM rheo-diecast parts exhibit distinctly different microstructures featuring refined primary α-Mg with rosette-like or near-spherical morphologies, along with fine secondary solidification structures (α_2-Mg).

[Figure 4: see original paper] shows the average size and shape factor of primary α-Mg grains in AZ91D alloy rheo-diecast parts at different rotation speeds with a barrel temperature of 560 °C. As evident from [Figure 3: see original paper] and [Figure 4: see original paper], increasing screw rotation speed leads to progressively finer and more spherical primary α-Mg grains with more uniform distribution. This is primarily attributed to two factors: First, higher rotation speeds intensify convection within the stirring chamber, enabling the melt to achieve greater undercooling within the same time period, which promotes nucleation. Simultaneously, enhanced convection creates more uniform temperature and concentration fields within the undercooled melt. Second, increased rotation speed substantially raises the frequency and intensity of collisions between dendrites and screw blades, between dendrites and chamber walls, and among dendrites themselves, facilitating dendrite arm fragmentation and rounding of irregular grain corners. However, excessively high rotation speeds should be avoided, as intense shearing and stirring can cause severe melt gas entrapment, resulting in numerous porosity defects within castings ([Figure 3: see original paper]d) that are detrimental to mechanical properties.

2.2 Effect of Barrel Temperature on Rheo-Diecast Microstructure

[Figure 5: see original paper] shows optical micrographs of AZ91D alloy FCM rheo-diecast tensile parts produced at a screw rotation speed of 500 r/min and barrel temperatures of 560–580 °C. [Figure 6: see original paper] presents the average size and shape factor of primary α-Mg grains at different barrel temperatures with a rotation speed of 500 r/min. As shown in [Figure 5: see original paper] and [Figure 6: see original paper], increasing barrel temperature results in larger primary α-Mg grain size, reduced grain number, more irregular morphology, and increasingly non-uniform distribution. This occurs because lower barrel temperatures increase the cooling rate of the melt within the stirring chamber, generating greater undercooling. This reduces the critical radius and critical work required for stable nucleation of primary grains, thereby enhancing nucleation rate and favoring the formation of numerous fine primary solid particles. Additionally, the higher number of primary grains increases the frequency of collisions and friction among solid particles during shearing, making the primary solid phase finer, more spherical, and uniformly distributed. Nevertheless, barrel temperature should not be too low, as excessively low temperatures severely impair slurry fluidity, creating difficulties for subsequent slurry transfer and diecasting. Therefore, selecting an appropriate barrel temperature is crucial.

2.3 Mechanical Properties of FCM Rheo-Diecast Parts

Table 1 summarizes the mechanical properties of AZ91D alloy diecast parts produced by different processes. Compared with traditional diecasting, FCM rheo-diecast parts show little change in yield strength but significant improvements in both ultimate tensile strength and elongation, which increase by 12.5% and 80.0%, respectively. This demonstrates the remarkable advantage of FCM rheo-diecasting in enhancing mechanical properties. The improvement stems from two main factors: First, the high viscosity and low temperature of semisolid slurry reduce gas entrapment during mold filling and solidification shrinkage, resulting in fewer internal defects. Second, as magnesium alloys have an hcp crystal structure that relies primarily on pyramidal twinning to coordinate basal slip for plastic deformation at room temperature, grain refinement and homogenization effectively alleviate local stress concentration and delay crack initiation and propagation.

In AZ91D magnesium alloy diecasting, non-equilibrium solidification causes brittle β-$Mg_{17}Al_{12}$ phase to distribute along α-Mg grain boundaries, which can be modified through heat treatment to improve mechanical properties. For traditional diecast parts, numerous internal pores cause local blistering during heat treatment. In contrast, FCM rheo-diecasting substantially reduces internal porosity, avoiding blistering during heat treatment and enabling further property enhancement through post-casting heat treatment. As shown in Table 1, T6 heat treatment yields the highest yield and ultimate tensile strengths (with ultimate tensile strength 8.9% higher than the as-cast condition) but lower elongation. T4 heat treatment produces the highest elongation (88.9% higher than as-cast) but lower yield strength. The as-cast condition exhibits the lowest ultimate tensile strength, with yield strength and elongation falling between those of T4 and T6 treatments.

[Figure 7: see original paper] shows SEM images of rheo-diecast tensile parts after T4 and T6 heat treatments. After T4 treatment ([Figure 7: see original paper]a), the eutectic phase distributed around primary solid grains gradually disappears, with most brittle β phase dissolving into the α-Mg matrix and grain boundaries becoming less distinct. This reduces the hindrance to dislocation motion from neighboring grains, decreasing yield strength. Solute atoms enter the matrix to form a supersaturated solid solution, weakening the embrittling effect of β phase on the matrix. With α-Mg as the dominant phase possessing good deformability, both tensile strength and elongation improve. After T6 treatment ([Figure 7: see original paper]b), the α-Mg solid solution undergoes discontinuous and continuous precipitation of β phase during aging. The precipitate morphology changes significantly compared with the as-cast structure, with β phase appearing as thin flakes and short rods with much smaller dimensions, which benefits strength enhancement. However, re-precipitation of β phase at grain boundaries during aging strengthens the hindrance to dislocation motion, reducing elongation.

To further explain the differences in mechanical properties, [Figure 8: see original paper] presents SEM fractographs of AZ91D alloy diecast tensile parts produced by different processes. The fracture surface of traditional diecast parts ([Figure 8: see original paper]a) exhibits typical cleavage fracture characteristics with numerous crystalline particles, showing primarily intergranular fracture and poor ductility. In contrast, FCM rheo-diecast parts ([Figure 8: see original paper]b) display quasi-cleavage fracture features with many small cleavage facets and river patterns formed by cleavage steps. The cleavage rivers are relatively disordered, with most cleavage facets connected by tear ridges, and most river patterns formed through tearing, indicating relatively better ductility. The T4 treated sample ([Figure 8: see original paper]c) shows tear ridges and obvious river patterns with numerous small dimples distributed around the river patterns, representing a typical quasi-cleavage fracture with brittle transgranular characteristics but good ductility. The T6 treated sample ([Figure 8: see original paper]d) exhibits typical cleavage fracture features with many rock candy-like and crystalline facets in random orientations, indicating poor ductility.

2.4 Microstructure Formation Mechanism and Solidification Behavior in FCM Rheo-Diecasting

In the FCM process, intense convection creates substantial bending stresses at dendrite roots. Since crystal strength is low near the melting point, mechanical dendrite fracture is possible, but this is not the primary mechanism for non-dendritic structure formation. The dendrite fragmentation viewpoint proposes that grains first grow as dendrites and then transform to spherical shapes under stirring action through fragmentation. This perspective evidently overlooks phenomena occurring during simultaneous stirring and solidification that differ from classical solidification theory.

The most significant characteristic of melt solidification under FCM conditions is that mechanical stirring generates intense mixed convection, and solidification proceeds under strong flow conditions, unlike the static solidification of conventional casting. The crystallization and solidification process is accomplished through nucleation and growth of crystals. The influence of FCM on dynamic crystallization primarily affects or even alters these two processes. The following analysis examines grain nucleation and growth during semisolid slurry preparation in the FCM device.

Under typical laboratory conditions, homogeneous nucleation does not occur in liquid metal solidification because the required undercooling is extremely large. Therefore, all grains in the FCM device are considered to nucleate heterogeneously. During semisolid slurry preparation, FCM action dramatically alters heat and mass transfer processes in the melt. Regarding heat transfer, after entering the FCM stirring chamber, the melt experiences strong cooling from both the barrel and stirring shaft, causing the temperature to drop below the liquidus within a short time. The entire melt achieves a uniform undercooled state rather than just the surface layer, enabling massive nucleation on suspended effective nucleation sites that can survive and continue growing. Regarding mass transfer, material transport in the melt is convection-controlled rather than diffusion-controlled, with the melt in a rapid mixing state. Solute rejected during grain growth is promptly removed without accumulation at the solid-liquid interface, maintaining relatively uniform macroscopic composition.

In addition to stable nuclei generated from effective nucleation sites within the melt, heterogeneous nucleation on the inner wall, screw, and rotating blades as substrates requires even less stable nucleation energy and more readily produces numerous stable nuclei. Ohno proposed a crystal dissociation mechanism when studying equiaxed grain origins, suggesting that necked grains form on mold walls or cooling liquid surfaces during early solidification, then detach and become free in the melt under convection ([Figure 9: see original paper]a). The FCM device operates based on this crystal dissociation mechanism, utilizing barrel and stirring shaft cooling combined with convective stirring and appropriate pouring temperature to enhance internal nucleation and promote grain detachment ([Figure 9: see original paper]b). Due to strong melt convection, these nuclei cannot form stable solidification shells and are washed into the melt interior while new nuclei continue forming on the walls, screw, and blades. This significantly increases the nucleation rate, creating conditions for crystallizing fine, spherical non-dendritic primary solid phases.

Once nuclei form, they enter the growth stage. When nucleus radius exceeds the critical value, growth proceeds spontaneously with decreasing system free energy. Intense convection alters the traditional solidification condition of unidirectional heat transfer by conduction and slow mass transfer by diffusion. Rapid mixing of heat and material within the melt creates relatively uniform temperature and composition throughout, placing grains in a uniform growth environment that substantially weakens dendrite formation conditions and enables uniform grain growth in all directions.

Mullins-Sekerka instability theory indicates that during solidification, when spherical crystals exceed a certain critical size, their stable growth becomes unstable and transitions toward dendritic development. Additionally, some nuclei in the undercooled melt grow in a dendritic manner on the barrel wall, screw, and rotating blades. However, strong convective stirring from the screw fragments these primary dendrites, which become free in the melt ([Figure 9: see original paper]b). Due to uniform temperature and concentration fields, free dendrites exhibit no preferred growth orientation. Simultaneously, under surface energy effects, free dendrites grow toward minimizing surface area, gradually becoming spherical. Molenaar et al. and Guo et al. proposed that grains under forced convection exhibit self-rotating behavior during growth, continuously altering the temperature and concentration fields at the liquid-solid interface. Therefore, even if significant growth rate differences appear among grain directions at any instant, grains grow relatively uniformly in all directions, ultimately producing spherical/near-spherical semisolid primary solid phases.

During the subsequent rapid slurry transfer stage, primary α-Mg enters a new growth phase characterized by relatively static conditions where morphology evolution occurs primarily under pure diffusion. From a solidification morphology perspective, grain spheroidization represents the result of solid-liquid interface morphology changes during transformation from unstable to stable states and subsequent stable maintenance. When slurry flows into the preheated collection crucible, self-stirring under gravity further homogenizes the temperature and concentration fields, allowing primary α-Mg sufficient time for spherical growth. Yang et al. identified the main factors affecting primary α-Mg morphology during rapid slurry transfer as the number of free crystals and slurry cooling intensity. Experimental results indicate that high crystal density and low cooling intensity favor spherical grain formation. In the FCM process, the preheated collection crucible provides low cooling intensity, while the slurry contains high-density primary α-Mg with short intergranular spacing. The concentration fields of neighboring grains overlap, further reducing the concentration gradient around each grain and diminishing destabilizing effects. This favors stable spherical growth, enabling free crystals generated by intense convection in the stirring chamber to continue growing to larger sizes in a near-spherical/spherical manner.

[Figure 10: see original paper] compares the solidification processes of AZ91D magnesium alloy melt in traditional diecasting and FCM rheo-diecasting. For FCM rheo-diecasting, AZ91D magnesium alloy melt solidification primarily involves two stages: primary solidification and secondary solidification. Primary solidification occurs during semisolid slurry preparation, comprising three phases: rapid melt cooling, isothermal stirring, and rapid slurry transfer. Secondary solidification includes solidification in the shot sleeve, mold cavity, and during mold filling—i.e., rheo-diecasting—which differs substantially from traditional diecasting. In traditional diecasting, melt nucleates first on shot sleeve and mold cavity walls due to undercooling. As the melt flows, some nuclei enter the interior, where some are remelted by superheated melt while others survive and grow. Under the influence of undercooling gradients, these nuclei mostly grow into dendrites with non-uniform distribution. In contrast, semisolid slurry used in rheo-diecasting contains numerous fine, uniform spherical crystals that mutually restrict growth during secondary solidification. With temperature in the semisolid range, undercooling readily induces nucleation and newly formed grains easily survive, resulting in final castings containing large quantities of fine, uniformly distributed near-spherical grains. Additionally, the high apparent viscosity of semisolid slurry reduces or eliminates turbulent flow during mold filling, decreasing casting porosity. Lower slurry temperature also reduces solidification shrinkage and improves internal defect conditions.

Since the shot sleeve and diecasting mold are made of heat-resistant steel with thick walls, low temperature, and strong heat absorption capacity, and the slurry contains limited heat with less latent heat to release, the cooling rate during secondary solidification in diecasting is much higher than primary solidification in the FCM device (which has graphite inner walls at 560–580 °C). Due to intense convective stirring, the residual liquid phase possesses uniform temperature and concentration distribution. Under strong cooling conditions, the undercooled residual liquid undergoes burst nucleation throughout without sufficient time for growth, ultimately forming fine secondary solidification structures (α_2-Mg).

Conclusions

1. FCM process parameters significantly affect the microstructure of AZ91D magnesium alloy castings; appropriately increasing screw rotation speed or decreasing barrel temperature benefits microstructure optimization.
2. Compared with traditional diecasting, FCM-processed AZ91D magnesium alloy rheo-diecast parts show little change in yield strength, while ultimate tensile strength and elongation increase by 12.5% and 80.0%, respectively. Heat treatment significantly affects mechanical properties of FCM rheo-diecast parts; compared with T4 and T6 treated parts, as-cast tensile parts exhibit the lowest ultimate tensile strength, with yield strength and

elongation falling between T4 and T6 conditions.

3. FCM action alters the traditional solidification condition of unidirectional heat transfer by conduction and slow mass transfer by diffusion, dramatically improving heat and mass transfer in the FCM device. Rapid mixing of heat and material creates relatively uniform temperature and concentration throughout the melt, placing grains in a uniform growth environment that destroys dendritic growth conditions and enables uniform grain growth in all directions.

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