Improving the microstructure and tensile properties of AZ91 magnesium alloy via electromagnetic stirring

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Abstract
In this study, the effects of electromagnetic stirring on the microstructure and tensile properties of AZ91 magnesium alloy were investigated by XRD, SEM, TEM, EBSD and tensile test. The results show that the yield strength, ultimate tensile strength and elongation increase to 167 MPa, 187 MPa and 7.5%, rising by 35.8%, 43.8% and 114.3% after the electromagnetic stirring, owing to the refinement of \( \alpha \)-Mg and \( \beta \)-Mg\(_{17}\)Al\(_{12}\) grains, the increased amount of low angle grain boundaries and the formation of a few tensile twins. The grain refinement mainly originates from the increase in nucleation rate. Electromagnetic stirring improves the plasticity of AZ91 alloy with poor ductility, and further increases the application of AZ91 alloy.

1. Introduction
Magnesium alloys are widely used in the automotive industry and 3 C production due to their excellent properties such as high specific strength and excellent casting properties [1–4]. Among the commercial magnesium alloys, AZ91 magnesium alloy (Mg-9Al-1Zn (wt%)) with superior machinability has been received great attention, in which the microstructure contains \( \alpha \)-Mg matrix and Mg\(_{17}\)Al\(_{12}\) phases [5, 6]. The ductility of AZ91 alloys is poor resulting from the limited slip systems in the hexagonal close packed (HCP) structure and coarse second-phase [7]. In order to improve the mechanical properties of AZ91 alloy, how to realize the grain refinement and the phase morphology and distribution becomes a particularly important issue.

Different techniques of grain refinement and phase transformation have been already used to enhance the mechanical properties. For the casting Mg alloys, the methods can be mainly classified into two categories. One method is the addition of rare earth elements such as Gd, Ce and Y into Mg matrix [8–11], but it greatly restricted due to higher cost and difficult control of casting qualities. Additionally, the microstructure of the alloy is improved by adding intermediate alloys and composite materials. The continuous network structure of AM60 alloy is destroyed by the addition of Mg-50Zn-5Y (wt%) intermediate alloy [12]. The B\(_4\)C particles and the matrix of the AZ91 alloy can be bonded well, but the addition of B\(_4\)C ceramic particles has little effect on the as-cast microstructure [13]. The molding process is another important method to improve the mechanical properties of the alloy, such as squeeze casting [14–16] and electromagnetic stirring (EMS) [17, 18].

Electromagnetic stirring technology improves the mechanical properties of the alloy mainly by the mean of microstructure refinement and temperature field homogenization, which derives form the electromagnetic force generated during solidification [19]. A reasonably homogeneous microstructure composed of fine grains with an average size of 45 \( \mu \)m is found by the EMS of AZ31 alloy [20]. The Mg particles are broken from dendritic to non-dendritic after EMS, the average primary size is refined from 680 \( \mu \)m down to 150 \( \mu \)m of Mg-2.5Gd-1Zn (at%) alloy [21]. The morphology of primary \( \alpha \)-Mg matrix phase in the Mg-3Nd-0.2Zn (wt%) alloy slurry is evolved from dendrite to rosette, dendrite, and spheroid after the EMS treatment [22]. However, these studies mainly focused on the Mg particle transformation, but the analysis of the phase transformation and texture...
evolution during EMS treatment usually is ignored. The purpose of this study is to investigate the effects of electromagnetic stirring with low frequency on the microstructure and tensile properties of casting AZ91 alloy, and the mechanisms of strengthening and toughening are revealed.

2. Experimental procedures

The chemical composition of the AZ91 alloy used in this study was shown in table 1. The commercial AZ91 alloy ingot was melted at 720 °C under a protective gas of N₂ and SF₆ during the melting process. The parameters of electromagnetic stirring were electric current and frequency with the values of 10 Hz and 150 A, respectively. The sample with electromagnetic stirring was denoted as modified sample. For comparison, in the same casting condition, a group of AZ91 alloys was prepared without electromagnetic stirring, and it was defined as unmodified sample. The molten metal was completely solidified under the work of magnetic stirrer, the sample of electromagnetic stirring was shown in figure 1.

![Sampling position](image1.jpg)

**Figure 1.** The sample obtained by electromagnetic stirring.

![Mandrel, Sleeve, Base](image2.jpg)

**Figure 2.** Three-dimensional diagram of the mould.

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The three-dimensional diagram of the mould was shown in figure 2, where the base and sleeve were graphite type, and the mandrel was sand type. A thermocouple was inserted at a distance of 42 mm from the inner wall of the sleeve and 60 mm from the base to measure the temperature evolution during the solidification. For the microstructure observation, the position of samples was chosen parallel to the direction of the magnetic field. To analyze the distribution and magnitude of magnetic force, the numerical simulation of electromagnetic field was performed using the Ansoft and Fluent software.

Tensile test analysis were carried out on the WGW-100H electronic universal testing machine with a strain rate of 0.001 s⁻¹ at room temperature, the tensile specimen was in accordance with 13239-2006 GB/T. The microstructure observation was conducted on by S-3400N scanning electron microscope (SEM), JEM-2010 transmission electron microscope (TEM), and electron back scattering diffraction (EBSD). EBSD analysis was
performed on a S-3400N scanning electron microscope equipped with a TSL electron backscatter diffraction analysis system. The SEM samples were mechanically grounded by SiC papers (grid 800#, 1200#, 2000#) and polished, then etched by acetic-picral solution (1 ml acetic acid, 0.5 g picric acid, 1 ml deionized water and 7 ml ethanol). The EBSD sample was ground to 50 μm, then it was electropolished by twin jet electro-polishing, and the electrolyte was 5% perchloric acid and 95% ethanol. The TEM sample was ground to 80 μm and then was prepared by ion thinning technique. X-ray diffraction analysis was carried out using XRD-7000 x-ray diffractometer of Cu-Kα with the scanning range of 20°–80° and the scanning speed of 2° min⁻¹.

3. Results

3.1. Microstructure

The phase constitution of AZ91 alloy is examined to understand whether any structural changes have taken place after electromagnetic stirring. Figure 3 shows the x-ray diffraction patterns of different AZ91 alloys. The XRD patterns reveal that the AZ91 alloy is mainly composed of Mg phase (PDF-#35-0821) and Mg₁₇Al₁₂ phase (PDF-#35-0821). The diffraction peaks appeared at 36.2°, 40.2°, 41.9°, 43.9° and 65.2° are the characteristic diffraction peaks of Mg₁₇Al₁₂ phase. After electromagnetic stirring, the XRD pattern shows that no other peak except for α-Mg matrix, and the diffraction peaks at 36.2°, 41.9°, 43.9° and 65.2° relative intensity of Mg₁₇Al₁₂ phase decrease compared to the unmodified sample, indicating that the volume fraction of Mg₁₇Al₁₂ phase in the alloy is reduced.

The SEM microstructures of the different AZ91 alloys are exhibited in figure 4. The microstructure of AZ91 alloy is composed of the Mg matrix and white second phases. The second phase is Mg₁₇Al₁₂ phase on basis of the XRD analysis in figure 3. The microstructure of the unmodified sample is shown in figures 4(a) and (b), which consists of an α-Mg matrix, β-Mg₁₇Al₁₂ phases and eutectic α + β phases. The eutectic α + β phases and the β-Mg₁₇Al₁₂ phases are mainly distributed on the grain boundary, the eutectic α + β phases are lamellar, and the β-Mg₁₇Al₁₂ phases are coarse dendrites. The constituents eutectic α + β phases are mainly the secondary β-Mg₁₇Al₁₂ phase precipitated from α-Mg solid solution.

The eutectic α + β phase is formed at the last stage of solidification, while the β-Mg₁₇Al₁₂ phase is generated after the end of solidification [23]. The microstructure of the modified sample shown in figures 4(c) and (d), the eutectic α + β phases are partially broken, the volume fraction of β-Mg₁₇Al₁₂ phases is reduced and broken up into small pieces. The distribution of β-Mg₁₇Al₁₂ phases become more uniform induced by electromagnetic stirring.

The TEM observation of AZ91 alloy is shown in the figure 5. There are some spherical particles that have an average size of about 80 nm, which is the Mg₁₇Al₁₂ phase determined by the inset diffraction pattern shown in figure 5(a). By comparison, the Mg₁₇Al₁₂ phase is further refined, and their distribution is more uniform after
The electromagnetic stirring, as shown in figure 5(b). The refined second phases with uniformity distribution is expected to optimize the mechanical properties of AZ91 alloy.

The EBSD maps and grain-size distribution of the AZ91 alloy are illustrated in figure 6. It can be seen that the grain size of the unmodified AZ91 alloy is not uniform, with a wide range from 15 μm to 200 μm, as shown in figure 6(c). However, after electromagnetic stirring, the grains become finer and more uniform, and the grain size is mainly distributed from 15 μm to 40 μm, as shown in figure 6(f).

The high angle grain boundaries (HAGBs) with misorientation larger than 10° are denoted by black line and the low angle grain boundaries (LAGBs) with misorientation within 2°–10° are labeled by white line. As shown in figure 6(a), the proportion of the misorientation angle less than 5° among grains is relatively high and the proportion of grain with a large misorientation angle of 15°–90° is more uniformed as shown in figure 6(b). As compared with the microstructure of unmodified specimen, the electromagnetic stirring significantly influences the microstructure. As shown in figure 6(d), a large number of grains with LAGBs and fragmented substructures combined with a few {1012} tensile twins appear within the grains of the modified specimen. During the electromagnetic stirring process, the twins are activated due to the force generated by the interaction between the solid phase and the liquid phase during the solidification. The distribution of the misorientation angle between adjacent grains has changed a lot after electromagnetic stirring. The HAGBs are mainly distributed between 80° and 90° as shown in figure 6(e), the misorientation angle distribution of 85°–90° existed in the sample because of the occurrence of twinning within the AZ91 alloy during the electromagnetic stirring process.

Figure 7 shows the textures of the different AZ91 alloys. The \{0001\}, \{1120\} and \{10\overline{1}0\} pole figures corresponding to EBSD maps are illustrated. The unmodified sample and modified sample exhibit a random texture. The maximum density of the unmodified sample is 12.27 as shown in figure 7(a), and the maximum
density is reduced to 6.79 after electromagnetic stirring as shown in figure 7(b). The decrease in the intensity of the basal texture is derived from the formation of fine grains.

3.2. Tensile properties
The stress-strain curves of the different AZ91 alloys are shown in figure 8. Under the same condition, the ultimate tensile strength and yield strength of unmodified sample are 130 MPa and 123 MPa, respectively, and the elongation is 3.5%. While the ultimate tensile strength of modified specimen reaches 187 MPa, yield strength increase to 167 MPa, and the elongation rises up to 7.5%, which increased by 43.8%, 33.1% and 114.3% as compared to the unmodified one respectively. It suggests that the strength and elongation of the AZ91 alloy are significantly improved after electromagnetic stirring.

Electromagnetic stirring improves the mechanical properties of the AZ91 alloy, and the addition of alloying elements can also improve the mechanical properties. Adding Sr and Ti elements can refine the grains of AZ91
alloy, the ultimate tensile strengths of adding 0.5 wt% Sr and 0.1 wt% Ti are 167 MPa and 174 MPa, respectively [24]. Adding Gd to AZ91 can significantly refine the \(\alpha\)-Mg grains and \(\text{Mg_17Al}_{12}\) particles, when adding 0.5 wt.% Gd, the ultimate tensile strength is 230 MPa [25]. In this paper, the ultimate tensile strength of electromagnetic stirred AZ91 alloy is 187 MPa, which is higher than the addition of Sr and Ti elements but lower than the Gd element.

There are some cleavage platforms and little ductile dimples in the fracture surfaces of the unmodified sample, and the size of cleavage platform varies from 60 \(\mu\)m to 200 \(\mu\)m, as shown in figure 9(a), indicating that brittle fracture plays a dominant role in the plasticity of unmodified sample. The fracture surface of the modified specimen is presented in figure 9(b). A number of fine cleavage platforms and some ductile dimples deriving from the fracture can be observed. There is a small tearing ridge on the fracture surface of the modified sample, which means that the alloy has plastic deformation during the fracture process [26]. When more ductile dimples and quasi-cleavage on the fracture surface occurs, the greater fracture energy leads to an increase in strength properties [27].

4. Discussion

According to the analysis mentioned above, after electromagnetic stirring, it is known that the mechanical properties of the alloy are improved due to the refinement of the crystal grains and the second phase. The grain size of the unmodified sample is uneven, while the grains of modified sample are finer and more uniform from the EBSD results. The full width at half maximum of the main crystal plane diffraction peaks of the Mg matrix is calculated by Jade software, as shown in table 2.

After electromagnetic stirring, the full width at half maximum increases. The grain size can be analyzed by the Debye–Scherrer formula.

\[
D = \frac{K\gamma}{B \cos 2\theta}
\]

Where \(K\) is the Scherrer constant, \(D\) is the average thickness of the crystal grains perpendicular to the crystal plane (nm), \(B\) is the value of the full width at half maximum of the diffraction peak (rad), \(2\theta\) is the diffraction
angle (in degrees), $\gamma$ is the x-ray wavelength (nm). According to equation (1), with the increases of $B$, the grain size decreases, further indicating that the grains are refined after electromagnetic stirring.

The grains are mutually inhibited when the alloy is deformed since the deformation resistance of grain boundaries is strong, thus the mechanical properties of the alloy are improved when the grains are refined [28]. According to EBSD results and full width at half maximum analysis, the grains are refined after electromagnetic stirring. Therefore, the enhanced strength of the alloy is attributed to the strengthening effects of grain refinement.

Grain refinement and phase fragmentation are caused by the generation of electromagnetic forces in a rotating magnetic field. According to Faraday’s Law, the electromagnetic force is proportional to the current. The current used in this study is 150 A. The magnetic flux density first reduces and then increases from the center to the edge, and according to the simulation result the magnetic flux density at the edge is about 0.4 T, as shown in figure 10. The microstructure of the material is generally related to the nucleation and growth conditions during solidification.

Figure 11 shows the cooling curves of different AZ91 alloys during the solidification. The temperature measurement point has a fast cooling rate of unmodified sample, with the cooling rate of around 2.04 °C s$^{-1}$. The cooling rate becomes slower under the electromagnetic stirring, with a value of approximately 1.83 °C s$^{-1}$.

Grain refinement is mainly due to the fact that electromagnetic force breaks the initial dendrites, the broken dendrites form new nucleation particles, which increases the number of the heterogeneous nucleation cites [18, 29]. Under the role of electromagnetic stirring, the primary $\alpha$-Mg phase nucleates at a relatively higher temperature, which increases the amounts of potential crystal nucleus. In addition, the broken dendrites also rotate while rotating with the melt convection, that eliminates the conformation of dendrites, and eventually grows into a spherical or near-spherical structure, thus the grains are refined [21].

According to the SEM and TEM observation, the electromagnetic stirring is found to play a specific role in the solidification process. In comparison with the microstructure obtained of specimen without the electromagnetic field, the eutectic network structures along the grain boundaries are finer and the $\beta$-Mg$_{17}$Al$_{12}$ phase is broken up into small particles, and the grain distribution becomes more uniform. On the other hand, after electromagnetic stirring, the phases located at the grain boundary of the alloy are reduced. This phenomena may occur because of the increased solubility of the solid alloy elements under the electromagnetic stirring during the solidification process, especially that of Al and Zn elements. Due to the strong mixed convection effect caused by electromagnetic stirring, the solute in the melt is rapidly mixed, which makes the composition of the melt more uniform [29].
The lattice constant of Mg is also analyzed, and the corresponding results are shown in Table 3. After electromagnetic stirring, the lattice parameters of \( a \) and \( c \) are decreased. When the ratio of the solute atomic radius of the solvent atomic radius is higher than 0.59 \[30\], the solute atom will occupy the position of the solvent atom to form a substitutional solid solution. The atomic radius of Al \((R_{Al})\) is 0.1432 nm, the atomic radius of Mg \((R_{Mg})\) is 0.1620 nm, and the ratio of \( R_{Al} \) and \( R_{Mg} \) is 0.88. Therefore, when the Al atoms are dissolved in the Mg matrix, the substitutional solid solution is formed in the AZ91 alloy, and the lattice parameters of \( a \) and \( c \) are reduced. The reduction of the lattice parameter indicates more Al atoms replace the Mg atoms on the original position.

The solubility of Al in the Mg matrix is increased by electromagnetic stirring, leading to the decrease of the precipitate along the grain boundaries. On the other hand, the second-phases are distributed dispersedly by the electromagnetic stirring. The refinement and discontinuity of the Mg\(_{17}\)Al\(_{12}\) phase also play an important role in the improvement of the ultimate tensile strength and elongation of the alloy.

Figure 12 shows the dislocation density in the crystallographic plane of \( \alpha \)-Mg matrix which calculated by the XRD patterns. The dislocation density can be obtained by the Dunn formula \[31\].

\[
\rho = \frac{L^2}{4.35 \times b^2}
\]  \( (2) \)

\( \rho \) is the dislocation density \((\text{cm}^{-2})\), \( L \) is the full width at half maximum \((\text{rad})\), and \( b \) is the Burgers vector. According to equation \(2\), four main slip planes, namely basal \((0002)\), prismatic \((10\overline{1}0)\), pyramidal \((10\overline{1}1)\) and \((10\overline{1}2)\) are calculated out. Dislocation density increases significantly on each crystallographic planes after electromagnetic stirring. The increase of dislocation density leads to a large stress concentration near the grain boundaries. In order to release the stress concentration, the dislocations are rearranged, so that the fraction of LAGBs increases \[32\]. As shown in figure 6, the fraction of the LAGBs from \(0^{\circ}\) to \(10^{\circ}\) increases rapidly. For the improvement of the mechanical properties of the alloy, the effect of the low angle grain boundary is the same as that of the high angle grain boundary, which mainly hinders the slip of the dislocation \[33\].

The improvement of strength and toughness of the AZ91 alloy after electromagnetic stirring is not only related to grain refinement, the fracture of the second phase and the increase of low angle grain boundary but also the generation of twins play an important role.

| Solidification condition | \( a \) (nm) | \( c \) (nm) | \( c/a \) |
|--------------------------|--------------|--------------|----------|
| Unmodified               | 0.3197       | 0.5199       | 1.6259   |
| Modified                 | 0.3195       | 0.5193       | 1.6256   |

The cooling curves of different AZ91 alloys during the solidification.

Figure 11. The cooling curves of different AZ91 alloys during the solidification.
5. Conclusions

(1) After electromagnetic stirring, the yield strength, ultimate tensile strength and elongation increase to 167 MPa (rising by 35.8%), 187 MPa (43.8%) and 7.5% (114.3%), respectively.

(2) After electromagnetic stirring, the grains are refined from the lamellar $\alpha + \beta$ eutectic phases and coarse $\beta$-Mg$_{17}$Al$_{12}$ dendrites on the grain boundaries. In addition, the solubility of Al in the Mg matrix increases.

(3) After electromagnetic stirring, a few $\{1012\}$ tensile twins formed and the fraction of refined grains with low angle grain boundaries increased. The presence of twins and low angle grain boundaries also play a role in the improvement of tensile properties.

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