

Fine Grain Microstructure of Mg-Al-Sr Alloy Solidified under High Pressures

Luyang Ren, Xuezhi Zhang, Li Fang, and Henry Hu

Abstract—In the present study, experimental work was performed to understand the solidification sequence and grain structure development of Mg-6wt.% Al-2wt.% Sr alloy (AJ62) under various applied pressures varying from 0 to 90 MPa. The analyses of recorded temperature histories indicate that the liquidus temperatures of the tested alloy during pressurized solidification increase when high pressures are exerted. The theoretical liquidus temperatures computed by the Clausius-Clapeyron equation deviate from the experimentally measured ones. The presence of the discrepancy suggests that, since it is almost impossible to experimentally measure local pressures in the cast alloy during solidification, the use of applied hydraulic pressures for the calculation of the theoretical liquidus temperatures should be responsible for the discrepancy. The microstructure analyses by optical microscopy show that the grain structures of the squeeze casting magnesium alloy AJ62 are refined with the application of pressures. The higher applied pressures result in the formation of the finer grains in the solidified alloy.

Index Terms—Applied pressures, grain refinement, Mg alloy, solidification, squeeze casting.

I. INTRODUCTION

Squeeze casting is a process which involves the solidification of a molten metal in a closed die under an imposed high pressure. Other terms used to describe the same or similar processes are liquid metal forging, extrusion casting and pressure crystallization [1], [2]. The high applied pressure which is several orders of magnitude greater than the melt pressure developed in normal casting processes keeps entrapped gases in solution and squeezes molten metal from hot spots to incipient shrinkage pores. As a result, the porosity in a squeeze cast component is almost eliminated. Furthermore, due to the minimization of the air entrapment at the liquid-mold interface by the applied high pressure, the heat transfer across die surfaces is enhanced, which increases solidification and cooling rates. Thus, superior mechanical properties of the casting resulting from the pore-free fine microstructure are achieved in squeeze casting processes [3]-[5]. The pressure as a primary parameter in squeeze casting has the most significant influence on a component via a variety of approaches which basically include changes in

solidification temperature of alloys and increases in the heat transfer rates across the casting/mold interface [6]. All these in turn affect the solidification behavior of a casting, which consequently manifests in the microstructure and mechanical properties of alloys. The nonequilibrium solidification occurring in squeeze casting could cause differences in microstructure between the actual solidified alloy and the prediction from phase diagrams.

To quantitatively analyze the influence of applied pressures on the liquidus temperature of an alloy system, the Clausius-Clapeyron equation can be employed as follows.

$$\frac{dT}{dA} = \frac{T_l \Delta V}{L_f} \quad (1)$$

where A is the applied pressure, T_l is the equilibrium liquidus temperature, L_f is the latent heat of fusion and ΔV is the volume change during phase change from liquid to solid. As the alloy freezes, ΔV and L_f are usually two minus terms owing to the alloy contraction and latent heat release, which lead to a plus value of $\frac{dT}{dA}$. Hence, an increase in applied

pressure results in a rise in the liquidus temperature of the alloy possessing a trend of volume decrease as it solidifies.

There are a lot of works in the literature on squeeze casting of Mg alloys. The influence of applied pressure, pouring and mold temperatures on the structure of squeeze cast Nd-containing Mg alloy was reported by Yang *et al.* [7] via process optimization. Yong and Clegg [8] discovered an applied pressure of 60 MPa was adequate to remove all minor shrinkage and air pores in the cast alloy, and the grain was refined from 127 to 21 μm whereas the pressure rose from 0.1 to 60 MPa. It was shown that the grain structure affected properties of squeeze cast Mg alloy AZ91 [9]. The study by Masoumi and Hu [10] demonstrated the influence of applied pressure (3 to 90 MPa) on the tensile behaviors and grain structure of squeeze cast AM50 alloy with Ca addition. Although numerous studies have been performed on squeeze casting of Mg alloys, most of them were primarily aimed at the development of relation between microstructure and properties. Research on microstructure of Mg alloys refined by applied pressures seems limited.

In the present work, thermal couples and pressure transducers were used to experimentally measure temperature and pressure variation during solidification of Mg alloy AJ62 in a cylindrical squeeze casting. The pressurized solidification including the liquidus temperatures are evaluated, and microstructure of Mg alloy AJ62 under various applied pressures is analyzed. The relationship between the liquidus temperature and grain refinement is identified and discussed.

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II. EXPERIMENTAL PROCEDURE

A. Alloy

Commercial Mg alloy AJ62 was employed in the work. Table I and Table II give the elemental percentages and thermophysical properties of the alloy. The alloy was commercially available and purchase from Magnesium Elektron Ltd, United Kingdom.

TABLE I: ELEMENT PERCENTAGE OF MG ALLOY AJ62

Alloy	Mg (wt. %)	Al (wt. %)	Mn (wt. %)	Sr (wt. %)
AJ62	balance	6.1	0.34	2.1

TABLE II: THERMOPHYSICAL PROPERTIES OF MAGNESIUM ALLOY AJ62

Material	Density ρ (g/cm ³)	Heat capacity C_p (kJ/kg·K)	Thermal conductivity k (W/m·K)	Solidus temp T_s (°C)	Liquidus temp T_l (°C)
AJ62	1.80	1.15	77	515	612

B. Alloy Preparation

Squeeze casting experiments were performed on a hydraulic press. A Lindberg (Lindberg/Blue M Thermo Electron Corporation Asheville, North Carolina, USA) electric resistance furnace was employed to melt magnesium alloy AJ62 held in a mild steel crucible with a maximum holding capacity of 4 kilogram. The furnace temperature was heated up and kept at 800°C. The melt temperature was regularly monitored by a digital thermometer (Omega HH509) during melting. The molten metal was poured directly from the crucible into the sleeve to minimize the loss of melt superheat once it reached a desired temperature. Protection gas (Sulfur Hexafluoride SF₆ 0.5% + carbon dioxide CO₂) was used during both melting and casting magnesium alloys to protect the melt from any excessive oxidation or possible burning.

A cartridge-based heating system was inserted in the upper and lower dies to preheat the top die to 250°C, and the lower die to 300°C. The liquefied alloy was introduced into the lower die at 680°C. The liquefied alloy was pushed and squeezed in the upper die by a desired applied pressure of 0, 30, 60, or 90 MPa, which was held for 25 seconds during freezing of the alloy. An illustration of the experimental setup is presented in Fig. 1(a). The final cylindrical coupon had a diameter of 0.10 m and a height of 0.03 m.

A Kistler pressure transducers 6175A2 (Kistler Instrument Corp., Amherst, New York, USA) were used in experiments to measure the cavity pressure. The sensor type 6175A2 consisted of a high temperature quartz sensor built into a rugged adapter. The sensor has a front diameter of 8 mm, is flush with the front of the adapter, separated by a cylindrical gap of <10 μ m, and measures the pressure directly. The pressure sensor was embedded in the upper die for recording the local pressure in real time as shown in Fig. 1(b). Simultaneously, during the squeeze casting process, local temperatures in the casting were measured by a K-type thermocouple, which was KTSS-116U-12 (Omega Engineering Inc., Stamford, Connecticut, USA). The KTSS-116U thermocouple molded assembled with transition joints of molded glass-filled nylon provide an economical yet durable thermocouple probe for a variety of

sensing applications. The measuring temperature was limited only by thermocouple type and sheath material. Stainless steel sheaths have a maximum temperature of 900°C, with Inconel 600 rated to 1150°C. Fig. 1(b) shows that the thermocouple was inserted in the casting center.

A data acquisition system contained a National Instrument (National Instruments Corporation, Austin, Texas, USA) PCI-6033E processor with eight temperature (K type) channels and eight sensor channels. During experiment, the analogue signals from thermocouples and sensors were fed to the data acquisition system through a National Instrument SCB-100 connector, and ultimately saved in a computer driver for permanent storage. A customized software was developed based on Lab View (a graphical development software by National Instruments Corporation, Austin, Texas, USA), which enables users to program and monitor the casting process. A LabVIEW-based data acquisition system was used to register all pressure and temperature measurements.

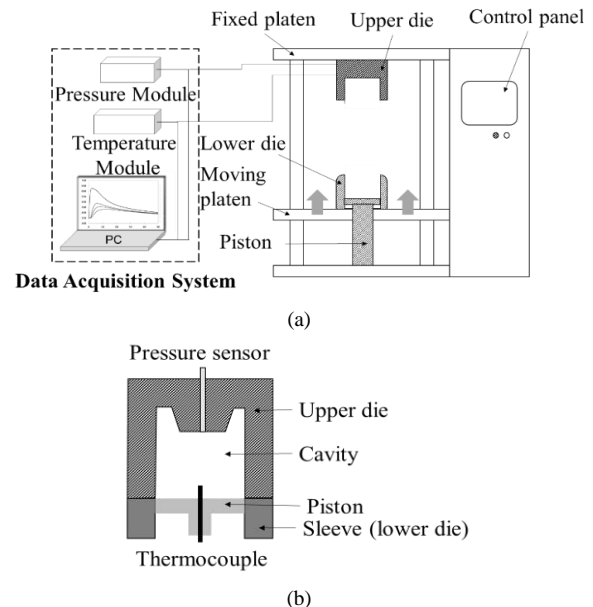


Fig. 1. Illustration of (a) the experimental setup and data acquisition system, (b) presence of pressure transducer and thermocouples in the die cavity.

C. Grain Structure Analysis

Samples were cut, mounted and polished from the center of the fabricated coupon and prepared following the standard metallographic procedures. To disclose the grain structure clearly, as-cast specimens were heat treated by a T4 treatment to dissolve the intermetallics (Mg₁₇Al₁₂). Based on the ASTM E112-96, grain sizes were measured by using an optical microscope.

III. RESULTS AND DISCUSSION

A. Pressure-Influenced Solidification Temperatures

Representative cooling curves recorded in the center of the squeeze casting solidified under applied hydraulic pressures of 0, 30, 60, and 90 MPa are given in Fig. 2. We can see from Fig. 2, that the temperature rose very fast right after the liquefied alloy was introduced into the sleeve of the lower die. Upon the application of the hydraulic pressure, an evident

increase in the liquidus temperature appeared. Subsequently, the temperature remained almost unchanged for a short period prior to the commencement of falling towards the solidus temperature. The increments in the liquidus and solidus temperature were consistent with the effect of applied pressures suggested by the Clausius-Clapeyron equation. When the applied pressure increased, both the liquidus and solidus temperatures rose. Furthermore, the observation on the cooling curves indicated that the change in applied pressures affected the holding time of the liquidus temperature plateau. With the applied hydraulic pressures of 0, 30, 60, and 90 MPa, the liquidus temperatures of the alloy appeared on the cooling curves were 608.38, 609.67, 610.23, and 612.19°C, respectively. The determined pressure-influenced liquidus temperature of the alloy was increased by 1.29, 1.85, and 3.81°C, when the applied hydraulic pressure became 30, 60, and 90 MPa, respectively. The total solidification time for each cast coupon was calculated based on the temperature data given by the cooling curves presented in Fig. 3.

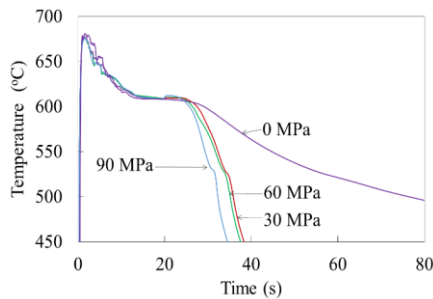


Fig. 2. Representative cooling curves of Mg alloy AJ62 squeeze cast under the applied pressures of 0, 30, 60, and 90 MPa.

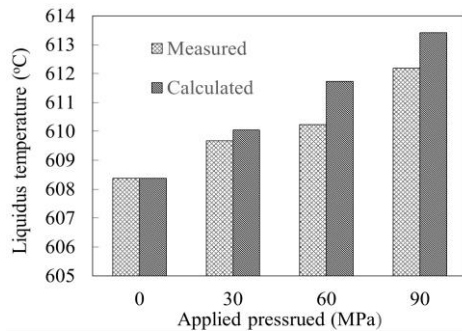


Fig. 3. Measured and calculated liquidus temperature of the Mg alloy AJ62 solidified under the applied pressures of 0, 30, 60, and 90 MPa.

The total solidification time was determined for the period in which the coupon solidified from its liquidus temperature to the end of the eutectic stop at the solidus temperature. When the alloy solidified under the atmospheric pressure (0 MPa), the solidus temperature was invisible on the cooling curves. In the case of the atmospheric pressure, the ending point of the solidification was set to be the instant as the temperature arrived at the calculated solidus temperature at 515°C. Examination of the determined solidification times revealed that, as the pressures of 30, 60, and 90 MPa were applied to the squeeze casting, the total solidification time were reduce by 29.5, 30.0, and 33.0 seconds in comparison with that (76.0 s) obtained under 0 MPa. The reduction in the total solidification time of the alloy was around 50% with the application of an external pressure over 30 MPa. Hence, the

rapid cooling of the alloy due to the application of a relatively large pressure was evident.

The effect of the applied pressures on both the measured and calculated liquidus temperature of Mg alloy AJ62 is illustrated in Fig. 3. The theoretical liquidus temperatures were calculated from Eq. (2) given below, which was integrated from Eq. (1):

$$T_i = T_m \exp\left(\frac{\Delta V}{L_f} A\right) \quad (2)$$

where T_i is the theoretical liquidus temperature under an applied pressure, T_m is the equilibrium liquidus temperature, ΔV is the volume change during solidification, L_f is the latent heat of fusion, P is the applied pressure. Compared to the equilibrium liquidus temperature of the alloy, there was an increase of 1.67, 3.34, and 5.02°C for the applied pressure of 30, 60, and 90 MPa, respectively. The discrepancy between the measured and theoretical temperatures could result from the inaccuracy in the input data of the hydraulic applied pressures in the integrated Clausius-Clapeyron equation, because it is impractical to insert the pressure transducer into the center of the liquid alloy to measure the local pressure, where the thermocouple was placed.

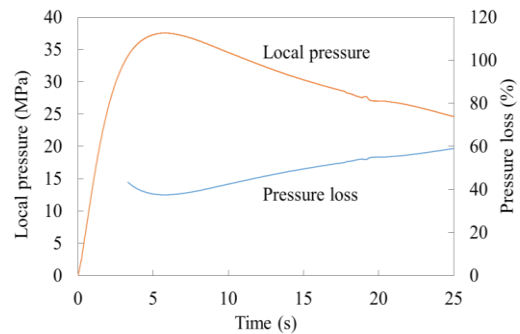


Fig. 4. Representative measured local pressure and pressure loss curves for Mg alloy AJ62 squeeze cast under the applied hydraulic pressure of 60 MPa.

B. Pressure Drop

The actual local pressures measured at the surface of the AJ62 cast coupon prepared under the hydraulic pressure of 60 MPa as a function of time is plotted in Fig. 4. The peak value of the measured local pressure was 37.54 MPa. The comparison of the measured local pressure and the applied hydraulic pressures indicated that there was a significant pressure drop by 22.46 MPa when the external pressure was applied during the solidification of the alloy. The local pressure loss was calculated by the expression below:

$$A_d (\%) = \left(\frac{A - A_p}{A}\right) \times 100 \quad (3)$$

where A_d is the pressure drop, A is the hydraulic pressure, and A_p is the peak value of experimentally measured local pressure. When the hydraulic pressure rose from 30 to 90 MPa, the pressure drop during solidification were 55.60, 37.43, and 31.85%, respectively. The pressure drop rose as the hydraulic pressure declined. This phenomenon of the pressure drop should be attributed to the alloy densification

and deformation caused by the application of the hydraulic pressure. The observation on the pressure drop gave an implication that, instead of using the local pressure, the use of the hydraulic pressure for the computation of the theoretical liquidus temperature could be responsible for the deviation between the computed results and experimental measurements (Fig. 3).

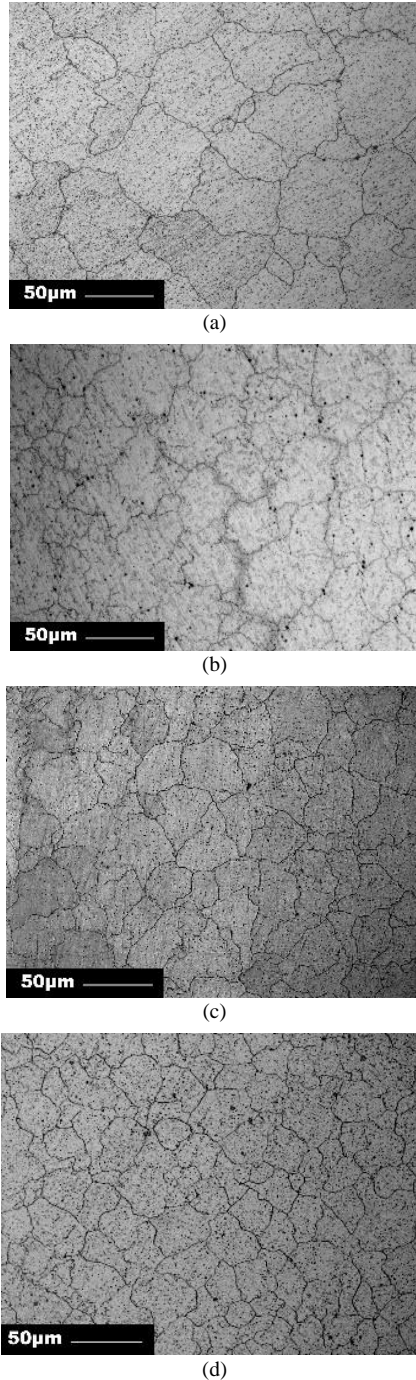


Fig. 5. Optical micrographs showing grain structures of the alloy prepared under the applied pressure of (a) 0 (b) 30 (c) 60 and (d) 90 MPa, respectively.

C. Grain Structure Refinement

Optical micrographs given in Fig. 5 reveals the grain structures of the Mg alloy AJ62 prepared under the applied pressures of 0, 30, 60, and 90 MPa. When the pressure rose from 0 to 30, 60, and 90 MPa, the average size of grains was reduced by 21, 29, and 36 μm , respectively, in comparison with 64 μm at 0 MPa. Fig. 6 presents the measurements of the

grain sizes. As illustrated in Fig. 3, since the applied high pressure caused a rise in the liquidus temperature of the alloy, the liquefied alloy was pressurized, which made solidified alloy be in tight touch to the mold surface for an extended period of time despite of its volume shrinkage owing to phase change from liquid to solid. The prolonged close contact between the alloy and the mold surface enhanced the heat extraction and promoted the rapid cooling, and as a result, led to the formation of the fine grain structure.

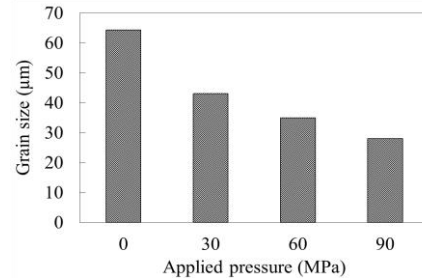


Fig. 6. Grain sizes vs the applied pressures of 0, 30, 60, and 90 MPa.

IV. CONCLUSION

The results of the present work indicate there are significant influences of the applied pressures on the solidification phenomena and grain structure of the Mg alloy AJ62 prepared by squeeze casting. The concluding remarks are summarized below:

- 1) Based on experimental measurements, an increase in the applied pressure results in a rise in the liquidus temperature of the alloy. There is an increase of 1.29, 1.85, and 3.81 $^{\circ}\text{C}$ in the liquidus temperature of the AJ62 when the hydraulic pressure increases to 30, 60, and 90 MPa from 0 MPa, respectively. In comparison with that (76.0 s) acquired under 0 MPa, there is a reduction of 29.5, 30.0, and 33 seconds in the total solidification time by the applied pressure of 30, 60, and 90 MPa, respectively.
- 2) A reduction in the applied hydraulic pressure leads to a local pressure drop. When the hydraulic applied pressure is 30, 60, and 90 MPa, the pressure drop percentage in squeeze casting of the alloy is 55.60, 37.43, and 31.85 %, respectively. The alloy densification and deformation caused by the applied pressure should be responsible for the occurrence of pressure loss.
- 3) The applied pressure increases the theoretical liquidus temperature of the alloy. The deviation between the measured and calculated results of the liquidus temperature should be caused by the inaccuracy in employing the local pressure value for the theoretical calculation, since it is impractical to insert the pressure transducer into the middle of the liquefied alloy to measure the local pressure, where the thermocouple was located. In the application of the Clausius-Clapeyron equation, the engagement of the hydraulic pressure value larger than the local pressure causes a slight overshooting in the calculation of the liquidus temperatures.
- 4) The high applied pressure refines the grain structure. The average grain size of the squeeze cast alloy is 64, 43, 35, and 28 μm at the applied pressure of 0, 30, 60, and 90

MPa. As a result, the grain size decreases by 33%, 45% and 56% when the pressure increases from 0 to 30, 60, and 90 MPa.

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