

Thixotropic behavior of semi-solid AZ91D magnesium alloy^①

MAO Wei-min(毛卫民), ZHEN Zi-sheng(甄子胜), YAN Shi-jian(闫时建), ZHONG Xue-you(钟雪友)

(School of Materials Science and Engineering,

University of Science and Technology Beijing, Beijing 100083, China)

Abstract: The thixotropic behavior of semi-solid AZ91D slurry was studied through a Couette type viscometer. The results show that the apparent viscosity of semi-solid AZ91D magnesium alloy slurry increases after being isothermally held, but the apparent viscosity quickly falls down to a steady state value after being stirred again and it takes on a sharp shear thinning behavior. With the same shearing rate and the rest time increasing, the steady apparent viscosity increases because of the agglomeration of the solid particles, and the time required for the slurry to reach the steady state also becomes longer. If the solid fraction increases, it takes longer time for the slurry to reach the steady apparent viscosity with the same shearing rate and the same rest time. If the solid fraction and the rest time are the same, but the shearing rate rises, it takes shorter time for the slurry to reach the steady apparent viscosity and the final steady apparent viscosity also decreases.

Key words: magnesium alloy; AZ91D; thixotropic behavior; semi-solid; apparent viscosity

CLC number: TG 146; O 373

Document code: A

1 INTRODUCTION

Magnesium alloys have been applied to the aeronautic, astronautic, mobile, motorcycle, electronic and information fields on a large scale because these alloys have small density, high specific strength and specific rigidity, good shock-resistance and machinability. During making machine parts with magnesium alloys, the semi-solid forming technologies are being used more and more^[1-6]. In order to control the semi-solid forming process and to manufacture better parts of magnesium alloys, it is necessary to realize the natures of the semi-solid magnesium alloy. The rheological and thixotropic behavior of semi-solid magnesium alloy are the basic natures. The rheological research has involved many alloys, but the thixotropic research of semi-solid metals is restricted on Sn-Pb and Al-Si alloys^[7-13] and the results relevant with the thixotropic behavior of semi-solid magnesium alloy has not yet seen in the published papers up to date. Therefore this paper has selected the AZ91D magnesium alloy as the test material and studied the thixotropic behavior of the semi-solid slurry of AZ91D by mechanical stirring.

2 EXPERIMENTAL

A Couette type viscometer was fabricated for the thixotropic testing and the schematic diagram is shown in Fig. 1. The stirring head with smooth sur-

face is assembled coaxially with the stirring crucible, and a narrow circular slot with 2.5 mm in width is formed between the stirring head and the

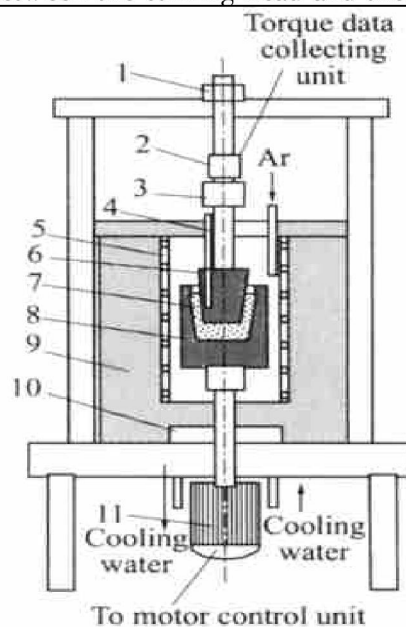


Fig. 1 Schematic diagram of experimental apparatus

- 1—Height adjuster; 2—Torque transducer;
- 3—Cooling water jacket; 4—Thermocouple;
- 5—Heating furnace; 6—Stirring head
- 7—Semi-solid slurry; 8—Crucible;
- 9—Heat insulation layer; 10—Cooling water;
- 11—Motor; 12—Protection gas

① **Foundation item:** Project(G2000067202) supported by the National Key Fundamental Research and Development program of China; project (G2002AA336080) supported by the National Advanced Materials Committee of China; project(50374012) supported by the National Natural Science Foundation of China

Received date: 2003 - 07 - 02; **Accepted date:** 2003 - 10 - 09

Correspondence: MAO Wei-min, Professor, PhD; Tel: + 86-10-62332882; E-mail: weiminmao@263.net

stirring crucible. The stirring crucible and stirring head are connected with a motor and a torque transducer respectively. By an accurate speed control unit, motor drives the crucible to rotate at a certain speed and a torque is produced on the stirring head because of the viscosity of the slurry. The torque transducer transforms the torque into electric signals. A computer with a data acquisition card then collected the torque electric signals and the torque can be transformed to the viscosity of the liquid or slurry using formula(1)^[14]:

$$\eta = \frac{M(R^2 - r^2)}{4\pi h \omega R^2 r^2} \quad (1)$$

where η is the viscosity of Newtonian liquid. As for non-Newtonian liquid, it should be named apparent viscosity; M is the torque on the stirring head; h is the immersed depth of the stirring head into the metallic liquid; ω is the angular speed of the motor; R is the inner radius of the crucible and r is the radius of the stirring head.

The average shear rate $\dot{\gamma}$ produced on the liquid can be calculated by formula(2)^[15]:

$$\dot{\gamma} = \frac{2\omega Rr}{R^2 - r^2} \quad (2)$$

The magnesium alloy was firstly placed in the crucible and melted by the resistant furnace outside. Then the liquid magnesium alloy was cooled continuously and was stirred by mechanical device simultaneously while a data detecting system was opened and

the viscosity data were recorded. When the magnesium alloy slurry reached the given temperature in the semisolid region, the cooling was stopped, and the slurry was kept isothermally and meanwhile it was stirred continuously. As the apparent viscosity of the semisolid slurry reached the steady value, the stirring was stopped. Because the starting semisolid slurry came from the above processing and was required to be in the same state, the same cooling rate and shearing rate might be maintained in making the starting slurries. Therefore, the continuous cooling rate of 4 °C/min and the shearing rate of 93.7 s⁻¹ were selected to make these starting slurries. After the semisolid starting slurry was kept static for some time, it was stirred again at the shearing rates of 93.7, 374.8 and 499.8 s⁻¹ respectively and the apparent viscosity of the slurry was detected.

3 RESULTS AND DISCUSSION

Under the condition of 580 °C (equal to $f_s = 0.34$) and 570 °C (equal to $f_s = 0.48$) and different resting times, such as 5, 30 and 60 min, the curves, which were detected from the slurry sheared once again at different shearing rates of 93.7, 374.8 and 499.8 s⁻¹, between the apparent viscosities of the semisolid slurry and the stirring time are shown in Fig. 2. It can be seen from Fig. 2 that the apparent viscosity of AZ91D magnesium alloy slurry increases after

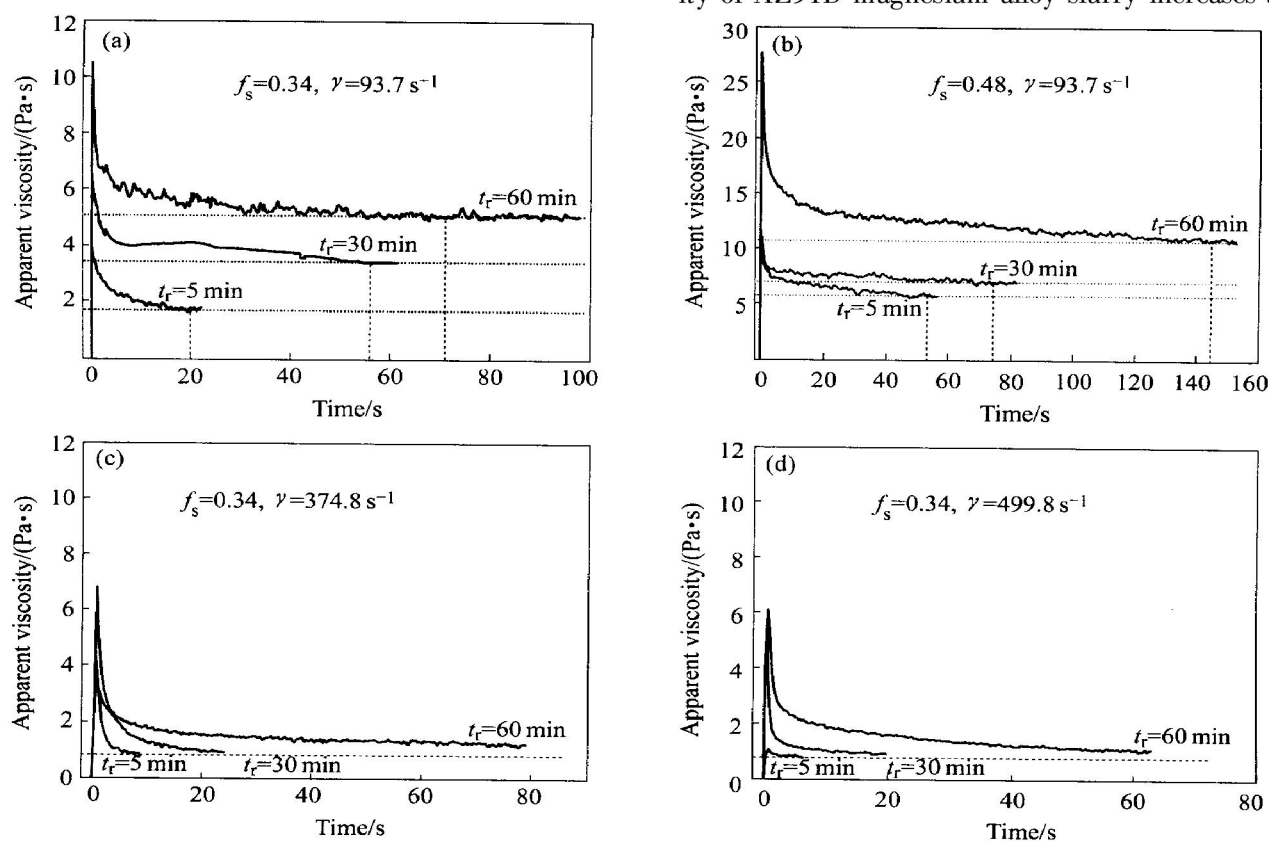


Fig. 2 Thixotropic behaviors of semisolid slurry of AZ91D magnesium alloy with different resting times

being kept static for some time, but decreases sharply as the shearing effect is added on the slurry again. The apparent viscosity eventually comes to a steady value. These curves also show that the slurry has specific shear-thinning behavior and that the resting time, shearing rate and solid fraction all have effect on the thixotropic behavior.

If the resting lasts for different times, the apparent viscosity of non-dendritic semi-solid slurry of AZ91D magnesium alloy will have different characteristic as the stirring is put on the slurry once more. The longer the resting time, the larger the final steady viscosity is and a longer time is required for the slurry to get to the final steady viscosity, as shown in Fig. 3 and Fig. 4.

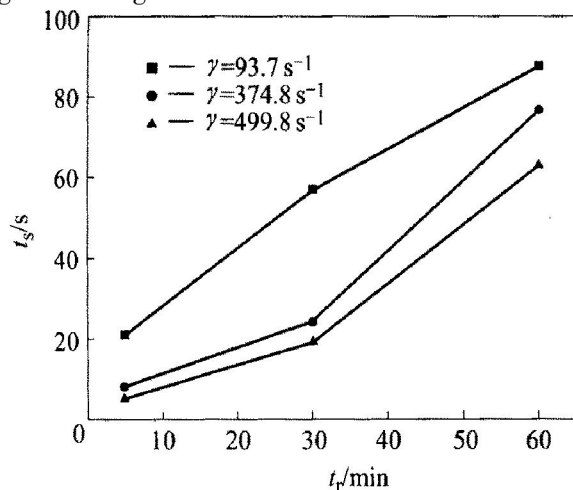


Fig. 3 Relationship of resting time with time required for slurry to reach steady state

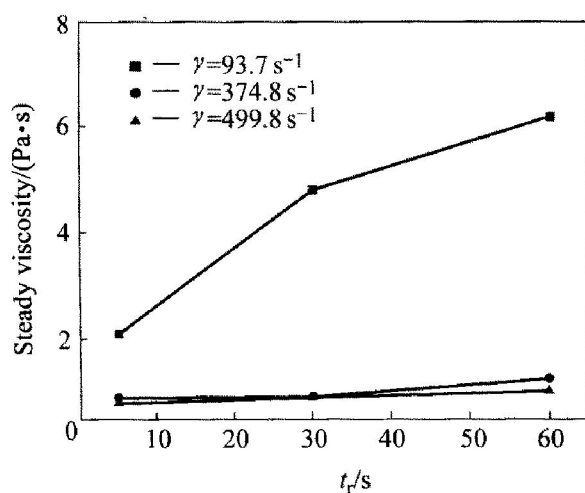


Fig. 4 Relationship of resting time with steady apparent viscosity

As to semi-solid metal slurry, the ideal microstructural characteristic should be that the primary grains of the slurry are spherical and evenly stretched in the residual liquid like an even suspension system. However, the slurry always regulates spontaneously its microstructural state according to the lowest sys-

tem energy law when being kept static. With regard to AZ91D magnesium alloy slurry system, it is found from observing the microstructure that some primary grains spontaneously agglomerate each other and are even combined together. That is agglomeration process. The agglomerated primary grains are larger in size and irregular in shape. So the slurry flow resistance increases and that is the main reason why the apparent viscosity rises to a higher value after the slurry is rested for some time. If the slurry with some extent of agglomeration is stirred again, the agglomerated primary grains will be destroyed by the shearing force and an even slurry system is formed again so that the apparent viscosity decreases with shearing time increasing. This phenomenon is called de-agglomeration process or shear-thinning process.

Provided that the resting time is regarded as agglomeration time of the primary grains and the second stirring time as the de-agglomeration time of the agglomerated primary grains, it is found from the curves in Fig. 2 that the de-agglomeration process is much faster than the agglomeration process and the time required for de-agglomeration process is only one over tens of the time for agglomeration process. The de-agglomeration process and the agglomeration process are mainly determined by their physical natures. Although the physical essence of the agglomeration process of primary grains is not all clear now, one aspect about the process is obviously understood, that is, the agglomeration of the primary grains kept in static state is completed through the micro-thermal motion. However, the de-agglomeration process is much more different from the agglomeration process and it is completed in the slurry flow under the external force condition. The forced flow motion and the vigorous collision between the primary grains compel the agglomerated grains to be destroyed. Therefore, the de-agglomeration speed is much higher than that for agglomeration. The longer the resting time, the more thorough the agglomeration extent between the primary grains is and the higher the apparent viscosity is. When being stirred again, the time required for the agglomerated primary grains to de-agglomerate become longer and the time required for the semi-solid slurry to reach the final steady viscosity becomes longer too, as shown in the Fig. 2.

Along with the resting time increasing, not only the time required for the AZ91D magnesium alloy slurry to reach the steady state become longer, but also the final steady viscosity become larger. This is mainly because the primary grains are getting to be larger for agglomeration and especially for combination among them when being kept static isothermally, as shown in Fig. 5. Several primary grains become one grain because of the

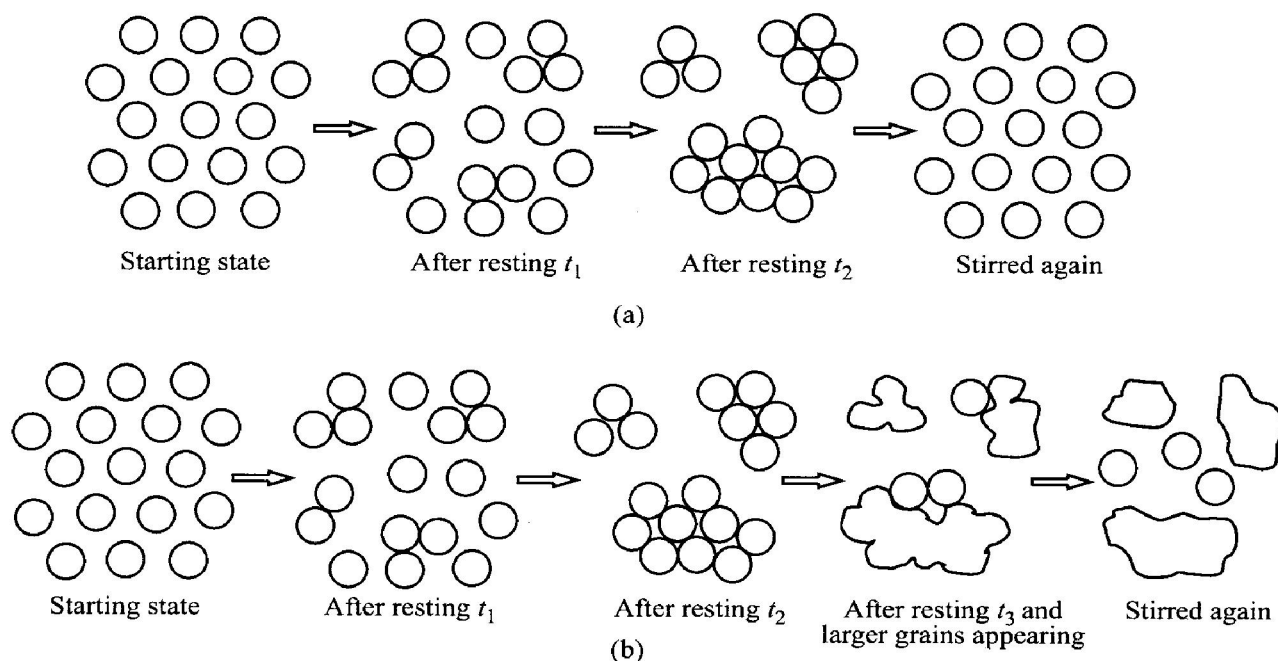


Fig. 5 Schematic of agglomeration and de-agglomeration between primary grains in semi-solid slurry ($t_1 < t_2 < t_3$)

- (a) — Agglomeration and de-agglomeration process in ideal state;
 (b) — Agglomeration and de-agglomeration process in this experiment

combination process. If the slurry is stirred again, the primary grains cannot be thoroughly recovered to their initial state, which promotes the flow resistance of the slurry and the final steady viscosity value. The longer the resting time, the more completely the combination among the primary grains happens and the more difficult the recovery of the primary grains to their initial state is. As a result, the final steady viscosity becomes larger.

Comparing Figs. 2(a), (c) and (d), it is also found that the semi-solid slurries of AZ91D magnesium alloy with the same solid fraction have displayed different thixotropic behaviors when being stirred again at different shearing rates. If the resting time is the same, the time required for the slurry to reach the steady state decreases with the shearing rates increasing and meanwhile the steady viscosity decreases too. According to the mechanism of agglomeration and de-agglomeration, the higher the shearing rate is, the faster the de-agglomeration is completed and the shorter the time required for the slurry to reach the steady state is. For the same reason, the de-agglomeration of the agglomerated primary grains is completed more thoroughly and the primary grains are stretched more evenly, and moreover, the agglomerated primary grains also become rounder at higher shearing rate. So these facts make the final steady apparent viscosity decrease.

Comparing Figs. 2(a) and (b), it is also found that the semi-solid slurries of AZ91D magnesium alloy with a high solid fraction need longer time to reach

the steady state than that with a low solid fraction. This is mainly because there are more primary grains in the slurry with a higher solid fraction and it is more likely for the primary grains to agglomerate when being kept static isothermally. Consequently the longer time is required for the slurry to complete the de-agglomeration.

4 CONCLUSIONS

1) The apparent viscosity of semi-solid AZ91D magnesium alloy slurry increases after being held isothermally, but the apparent viscosity quickly falls down to a steady state value after being stirred again and it takes on a sharp shear-thinning behavior.

2) With the same shearing rate and the rest time increasing, the steady apparent viscosity increases because of the agglomeration of the solid particles, and the time required for the slurry to reach the steady state also becomes longer.

3) If the solid fraction increases, it takes longer time for the slurry to reach the steady apparent viscosity with the same shearing rate and the same resting time.

4) If the solid fraction and the resting time are the same, but the shearing rate rises, it takes shorter time for the slurry to reach the steady apparent viscosity and the final steady apparent viscosity also decreases.

REFERENCES

- [1] Bradley N L, Weiland R D, Achafer W J, et al. US Patent: 5040589, 1989.
- [2] Frederick P S, Bradley N L, Erickson S C. Injection molding magnesium alloys[J]. *Advanced Materials & Processes*, 1988, 134(4): 53 - 56.
- [3] Decker R F, Carnahan R D, Vining R, et al. Progress in thixomolding[A]. Kirkwood D H, Kapranos P. *Proc of the 4th Int Conf on Semisolid Processing of Alloys and Composites*[C]. UK: Department of Engineering Materials, University of Sheffield, 1996. 221 - 225.
- [4] Leng T. Thixosystem[A]. Chiarmetta G L, Rosso M. *Proc of the 6th Int Conf on Semisolid Processing of Alloys and Composites*[C]. Turin, Italy, Materials Science and Chemical Engineering Department, Politecnico DI Torino: 2000. 215 - 220.
- [5] Peng Hs, Hsu W M. Development on rheomolding of magnesium parts[A]. Chiarmetta G L, Rosso M. *Proc of the 6th Int Conf on Semisolid Processing of Alloys and Composites*[C]. Turin, Italy, Materials Science and Chemical Engineering Department, Politecnico DI Torino, 2000. 313 - 317.
- [6] Ji S, Fan Z, Liu G, et al. Twin-screw rheomolding of AZ91D Mg alloy[A]. Tsutsui Y, Kiuchi M. *Proc of the 7th Int Conf on Semisolid Processing of Alloys and Composites*[C]. Tsukuba Japan, National Institute of Advanced Industrial Science and Technology, Japan Society for Technology of Plasticity, 2002. 683 - 688.
- [7] Flemings M C. Behavior of metal alloys in the semisolid state[J]. *Met Trans*, 1991, 22A(5): 957 - 981.
- [8] LI Y J, MAO W M, ZHEN Z S, et al. Microstructure characteristics and apparent viscosity of hypereutectic Al-24%Si alloy melt during semisolid state stirring[J]. *J Uni Sci & Tech Beijing (English edition)*, 2001, 8(2): 126 - 128.
- [9] Kattamis T Z, Piccone T J. Rheology of semisolid Al-4.5% Cu-1.5% Mg alloy[J]. *Mater Sci Eng*, 1991, 131A(2): 265 - 272.
- [10] Quaak C J, Horsten M G, Kool W H. Rheological behaviour of partially solidified aluminum matrix composites[J]. *Mater Sci Eng*, 1994, 183A(1 - 2): 247 - 256.
- [11] Azzi L, Ajersch F, Stephenson T F. Rheological characteristics of semisolid GrAlNi composite alloy[A]. Chiarmetta G L, Rosso M. *Proc of the 6th Int Conf on Semisolid Processing of Alloys and Composites*[C]. Turin, Italy, Materials Science and Chemical Engineering Department, Politecnico DI Torino, 2000. 527 - 532.
- [12] Young K P, Riek R G, Flemings M C. Structure and properties of thixocast steel[J]. *Metals Tech*, 1979, 6(4): 130 - 137.
- [13] ZHEN Zisheng, MAO Weimin, YAN Shijian, et al. The apparent viscosity of semisolid AZ91D alloy at steady state[J]. *Acta Metalurgica Sinica(English Letters)*, 2002, 15(6): 505 - 510.
- [14] Spencer D, Flemings M C. Rheological behavior of Sn-15%Pb in the crystallization range[J]. *Metall Trans*, 1972, 3(7): 1925 - 1932.
- [15] Joly P A, Mehrabian R. The rheology of a partially solid alloy[J]. *J Mater Sci*, 1976, 11(8): 1393 - 1418.

(Edited by PENG Chao-qun)