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Steady state rheological behavior of semi-solid ZK60-RE magnesium alloy during compression

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Abstract: Steady state rheological behavior of semi-solid ZK60-RE magnesium alloy during compression was studied. The alloy was prepared from ZK60 alloy and RE elements by casting, equal channel angular extruding, and liquidus forging. Semi-solid isothermal pre-treatment was carried out to make the grains spherical before compression. The apparent viscosity increases with decreasing the solid content and shear rate. Another very important factor is the grain size. When the solid content is high, the viscosity increases with decreasing the grain size at high strain rates and decreases with decreasing the grain size at low shear rates. Several fitting equations were obtained by using the power law equation, and the method of time-temperature superposition was used to get more information through a small number of experimental data.

Key words: ZK60-RE magnesium alloy; rheological behavior; method of time-temperature superposition; fitting equation

1 Introduction

Magnesium alloys have high potential as structural components in automobile and electronic device applications because of their low density and high strength. Now, casting magnesium alloys are mainly used for these purposes through die-casting or thixomoulding. In order to achieve more substantial structural applications of magnesium alloys, it is necessary to develop wrought magnesium alloy products such as ZK60 magnesium alloy. However, magnesium alloys have low tensile strength, poor ductility and low corrosion resistance compared with aluminum alloys. It has been reported that rare earth(RE) additions can improve casting characteristics, corrosion resistance, high temperature tensile strength and ambient tensile yield strength of as-cast magnesium alloys[1−2].

Semi-solid process of magnesium alloy is a kind of new technology, whose characteristics are as follows [3−6]: 1) reducing the porosity and continuous pores; 2) reducing the residual stress; 3) improving the flatness and dimensional stability; 4) improving the heat resistance and extensibility. In order to improve the quality of semi-solid magnesium alloy products and the accuracy of computer simulation in semisolid process of magnesium alloy, it is necessary to study the rheological characteristics of semisolid magnesium alloy. Much similar work has been reported. The steady state rheological behaviour of semi-solid A356 and A357 aluminum alloys were researched[7−9], and the results showed that the apparent viscosity increases with increasing the solid fraction but decreases with increasing the shear rate in the steady state. The steady state rheological behaviour of semi-solid AlSi4Mg2 and AlSi6Mg2 alloys were also researched[10−11]. The results showed the apparent viscosity of semi-solid AlSi6Mg2 alloy at steady state increases with increasing solid fraction, but declines with increasing shearing rate. The steady state rheological behavior of semi-solid AZ91D magnesium alloy was researched by GEBELIN et al[12] and JIANG and LUO[13]. The results showed that the magnesium alloy has a clear shear-shinning characteristic. Although some researches about the rheological behavior of semi-solid alloys were reported, rather less attention was paid to ZK60-RE magnesium alloy and the fitting equations. In this study, steady state rheological behavior of semi-solid ZK60-RE magnesium alloy during compression is researched; and the fitting equations and more information about rheological

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behavior are researched as well.

2 Experimental

There were three kinds of preparation method in experiments including casting, equal channel angular extruding(ECAE) and liquidus forging ZK60-RE magnesium alloys. These alloys were machined to cylindrical specimens of d 8 mm×10 mm for the compressing experiments. A Gleeble-1500D simulator was used in compressing experiments. Fig.1 shows the schematic diagram of compression. A thermal couple was embedded in a hole drilled on the specimen before compression. Each specimen was placed between two parallel blocks and heated to the desired temperature, and held for certain time at that temperature, followed by compression. Then the specimen was taken out and quenched in water. The liquid graphite was painted on the compressing surface to reduce the friction. The heating rate was 5 ℃/s.

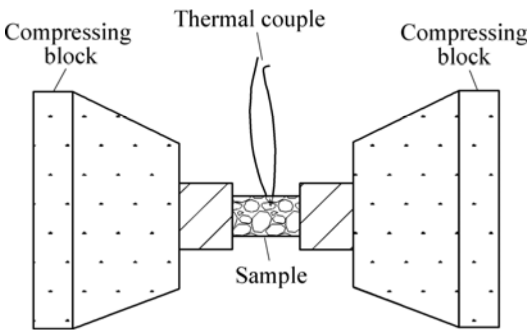

Fig.1 Schematic diagram of compression

The experiments were carried out under different conditions according to different varied factors.

1) Varying the solid fractions by means of varying the temperature. Here, the semi-solid temperature selected was 550, 560 and 570 ℃, and the corresponding solid content was 0.87, 0.84 and 0.8 respectively according to the calculation with MATLAB software. In these groups, the alloy used was casting ZK60-RE magnesium alloys. Small specimens were heated to 600 ℃ (the corresponding solid fraction is 0.67) and held for 7 min before compression to ensure the spheroidal and uniform microstructure.

2) Varying the holding time. In this group, the used alloy was ZK60-RE magnesium alloy prepared by ECAE. Specimens were held for different time: 3, 7, 10 and 15 min at semi-solid temperature before compression. The semi-solid temperature was 560 ℃ and the strain rate was 1 s^{-1}.

3) Varying the strain rate. The selected strain rates were 1×10^{-2}, 1×10^{-1}, 1 and 10 s^{-1}. These experiments included two groups. The first group used casting ZK60-RE magnesium alloys at the temperatures of 550, 560 and 570 ℃, and the second group used all the three kinds of ZK60-RE magnesium alloys at the temperature of 560 ℃.

4) Varying the grain size. Some relative experiments were done to show the different grain size of three kinds of ZK60-RE magnesium alloys. Grain size is important to influence the mechanical properties of semi-solid parts, originally because it influences the mechanical behavior. In this group, three kinds of ZK60-RE magnesium alloys were researched because of their different grain size. The temperature was 560 ℃.

3 Results

Apparent viscosity is a unique criterion to describe the rheology of semi-solid material and it means the inner friction of the solid particles. From the compression experiments, a series of true stress—true strain curves under different conditions can be obtained, and these results can reflect the rheological characteristic by the apparent viscosity η_a and the shear rates $\dot{\gamma}$ calculated by using Eqns.(1) and (2)[12].

$$\eta_a = \frac{2\pi h_\varepsilon^4}{3V^2\dot{\varepsilon}}F_\varepsilon \tag{1}$$

$$\dot{\gamma} = \frac{1}{2}\sqrt{\frac{V}{\pi h_\varepsilon^3}}\dot{\varepsilon} \tag{2}$$

where V is the volume of sample, $\dot{\varepsilon}$ is the strain rate.

3.1 Variation of apparent viscosity with temperature

Temperature is a very important factor influencing the apparent viscosity. Fig.2 shows the variation of apparent viscosity of casting samples with temperature under different shear rates. The holding time was 0.5 min. From Fig.2, with increasing temperature, hence increasing the liquid fraction, the apparent viscosity decreases. As the liquid fraction increases, there is a reduction of number of contacts among the particles, which reduces the inner friction among solid particles, resulting in the reduction of apparent viscosity.

3.2 Variation of apparent viscosity with holding time

Holding time is an uncertain factor influencing the apparent viscosity while it depends on the microstructure evolution during the holding time. Fig.3 shows the variation of the apparent viscosity with holding time for semisolid as-ECAEed ZK60-RE magnesium alloy compressed at various holding times. The strain rate was

1 s^{-1} and the temperature was 560 ℃. The microstructures shown in Fig.4 indicate that at 3 min and 7 min,

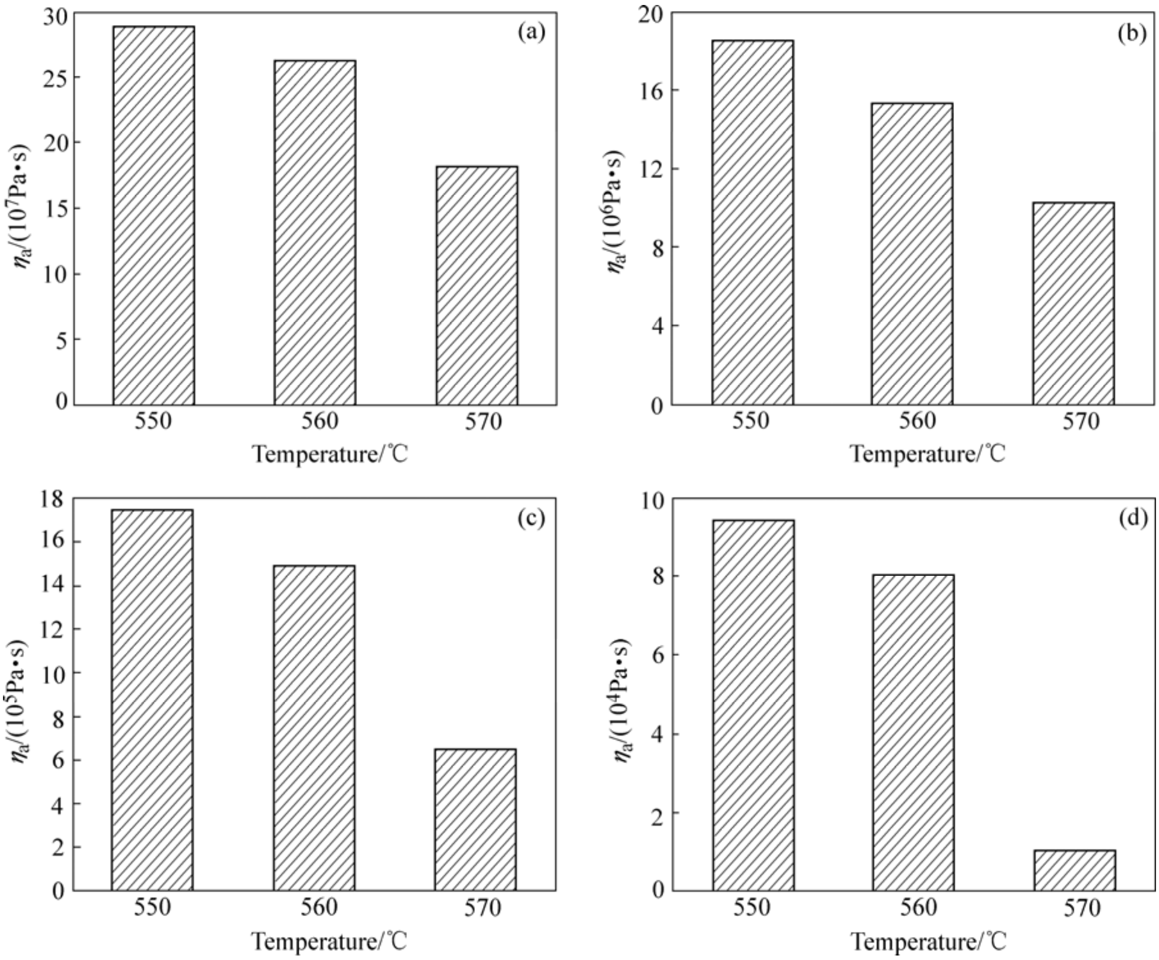

Fig.2 Variations of apparent viscosity with temperature for semi-solid ZK60-RE magnesium alloy: (a) $\dot{\gamma}$=6.59×10^{-3} s^{-1}; (b) $\dot{\gamma}$=6.59×10^{-2} s^{-1}; (c) $\dot{\gamma}$=6.59×10^{-1} s^{-1}; (d) $\dot{\gamma}$=6.59 s^{-1}

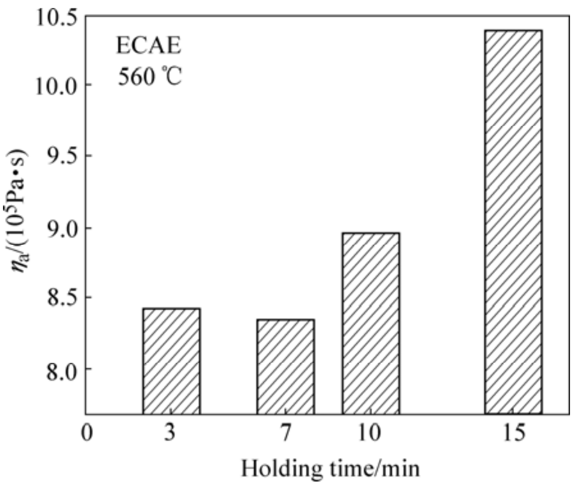

Fig.3 Variations of apparent viscosity with holding time for semi-solid ZK60-RE magnesium alloy

there is little variation in microstructures; hence the peak stress is similar. At 10 min, the solid particles have grown up and start to connect with each other, with liquid being enwrapped in the solid particles. This tendency is increased at 15 min. Therefore, with increasing the holding time, the apparent viscosity increases. Generally, holding time is a complex factor influencing apparent viscosity, because it influences the solid particle size, the shape factor and the degree of connectivity.

3.3 Variation of apparent viscosity with grain size

Fig.5 shows the variation of apparent viscosity with grain size during compression for semisolid ZK60-RE magnesium alloy prepared by three methods: casting, equal channel angular extruding and liquidus forging. At this solid fraction, the apparent viscosity increases with increasing the grain size. Fig.6 shows the microstructures of ZK60-RE prepared by three methods at 560 ℃ and holding time of 0.5 min. When the solid content is high, liquid can distribute uniformly among the solid particles (Fig.6(c)) and the connected necks among solid particles are less, while when the solid particles are coarse (Figs.6(a) and 6(b)) the liquid distributes nonuniformly, which results in the higher inner friction among solid particles, hence the apparent viscosity increases. This result may be contrary to some other results because the

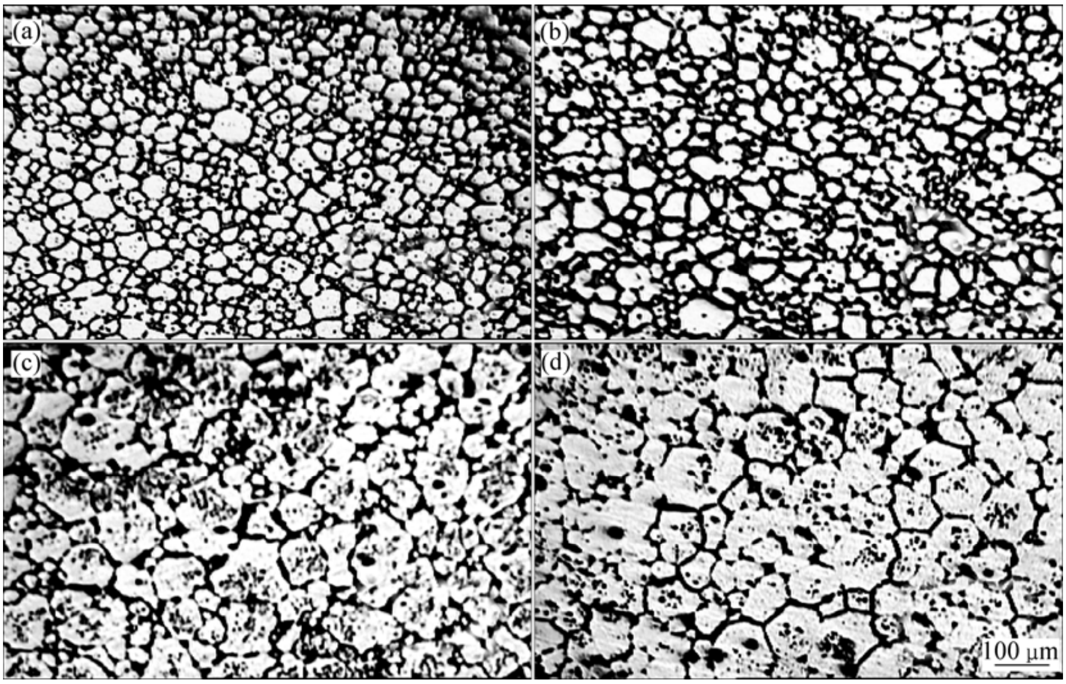

Fig.4 Microstructure evolution of as-ECAEed ZK60-RE magnesium alloy at different holding times: (a) 3 min; (b) 7 min; (c) 10 min; (d) 15 min

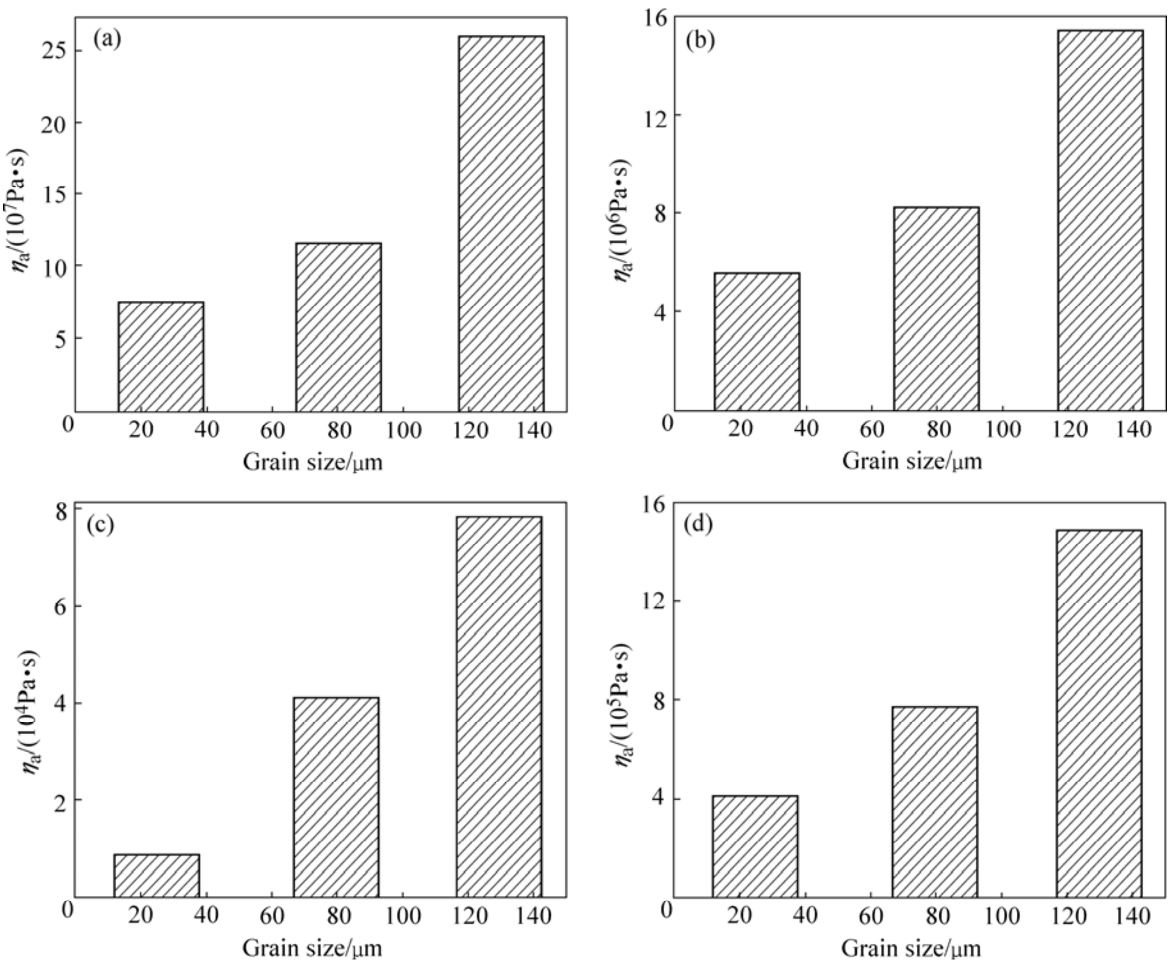

Fig.5 Variations of apparent viscosity with grain size for semi-solid ZK60-RE magnesium alloy: (a) 6.59×10^{-3} s^{-1}; (b) 6.59×10^{-2} s^{-1}; (c) 6.59×10^{-1} s^{-1}; (d) 6.59 s^{-1}

corresponding solid fraction and shape factor of grains are different. It has not stable rule but strongly depends on the microstructure.

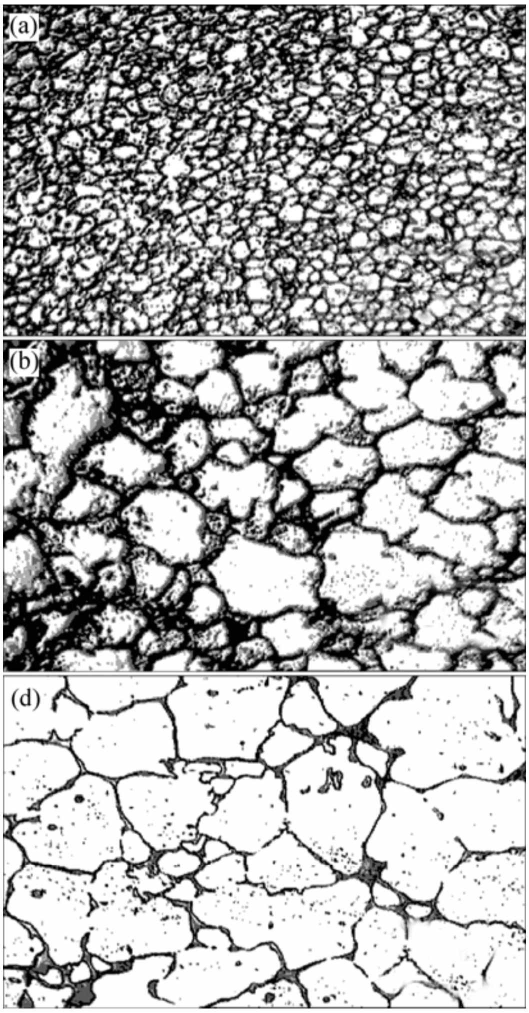

Fig.6 Microstructures of ZK60-RE magnesium alloy prepared by three methods at 560 ℃: (a) ECAE; (b) Liquidus forging; (c) Casting

3.4 Variation of apparent viscosity with shear rate

Shear rate is one of the most important factors to influence the apparent viscosity. Figs.7 and 8 show the variations of the apparent viscosity of semi-solid ZK60-RE magnesium alloy with shear rate. The apparent viscosity decreases obviously with increasing the shear rate. When the shear rate is 6.59×10^{-3} s^{-1}, the apparent viscosity is more than 10^8 Pa·s, while the apparent viscosity is 10^5 Pa·s when the shear rate is 6.59 s^{-1}. This shows a clear shear-shinning behavior of semi-solid ZK60-RE magnesium alloy. The shear rate will influence the behavior from two opposite ways. Increasing the shear rate will increase the possibility of particle-particle contact, but it will decrease the time of contact and the formation of a new solid-solid boundary is a time dependant process. Fig.9 shows the variation of viscosity with shear rate under different temperatures and different billets preparation methods, replotted in a log-log scale. The figure shows a clear pseudo-plastic behavior, and the curves are fitted to the power law equation[14]. Table 1 gives the fitting equation. The values of $n-1$ vary between −1.14 and −1.40.

From the experimental data, the values of $n-1$ are approximate; therefore, some useful equations are

Table 1 Fitting equations of apparent viscosity and shear rate

Material	Temperature/℃	Fitting equation
As-cast ZK60-RE	550	$\eta=10^{5.95248}\dot{\gamma}^{-1.15034}$
	560	$\eta=10^{5.88204}\dot{\gamma}^{-1.15834}$
	570	$\eta=10^{5.32329}\dot{\gamma}^{-1.39538}$
As-liquidus forged ZK60-RE	560	$\eta=10^{5.59429}\dot{\gamma}^{-1.14033}$
As-ECAEed ZK60-RE	560	$\eta=10^{5.16361}\dot{\gamma}^{-1.29936}$

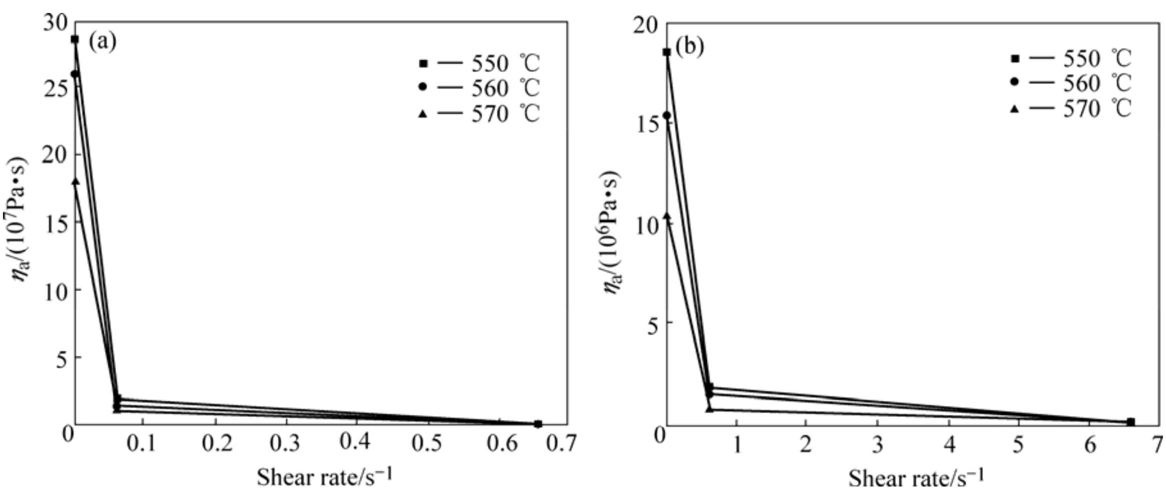

Fig.7 Variations of apparent viscosity with shear rate for semi-solid ZK60-RE magnesium alloy prepared by casting at different temperatures

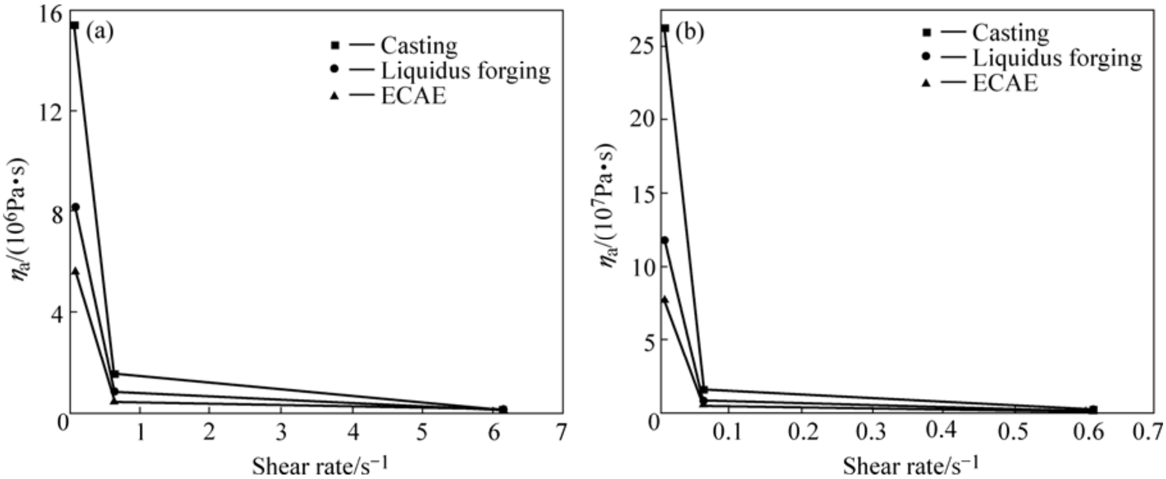

Fig.8 Variations of apparent viscosity with shear rate for semi-solid ZK60-RE magnesium alloy prepared at 560 ℃ by different methods

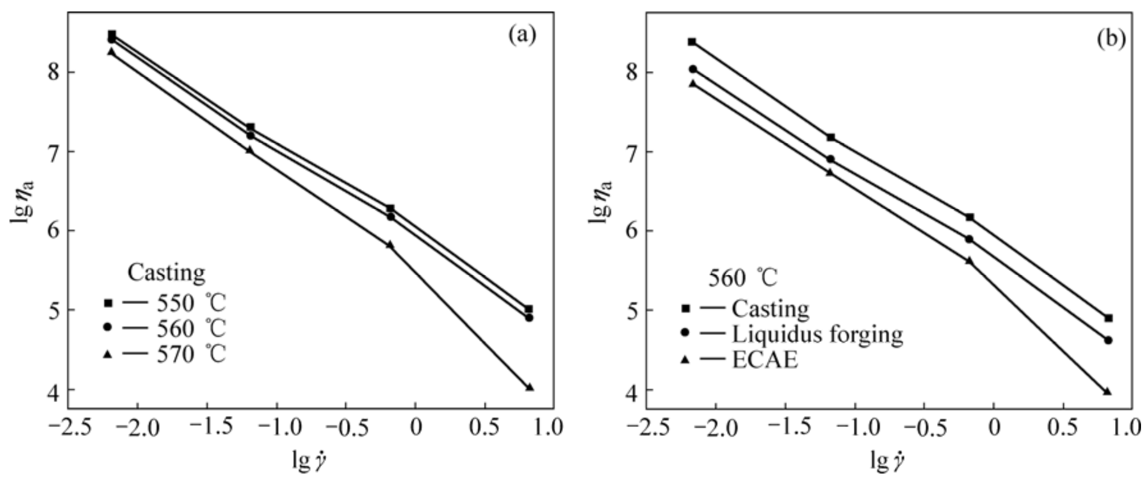

Fig.9 Variations of apparent viscosity with shear rate replotted in log form for semi-solid ZK60-RE magnesium alloy

obtained when these curves are supposed parallelly. The method of time-temperature superposition[15] can be used to describe the unknown curves since the shear rate is the reciprocal value of time. For the same alloys, we can choose a temperature to be the reference and its fitting equation of shear rate and viscosity can be obtained through experiments. Here, the fitting equation at this temperature is supposed to be

$$\eta = k\dot{\gamma}^{n-1} \tag{3}$$

Then we can get

$$\lg\eta = (n-1)\lg\dot{\gamma} + \lg k \tag{4}$$

In addition, if a viscosity value under a given temperature and a given shear rate is η_1, Eqn.(5) can be obtained according the same $n-1$:

$$\lg k_1 = (\lg\eta_1 - \lg\eta) + \lg k \tag{5}$$

Then the fitting equation of shear rate and viscosity under this given temperature can be written as

$$\begin{cases} \eta_1 = 10\exp[(\lg\eta_1 - \lg\eta) + \lg k]\dot{\gamma}^{n-1} \\ \eta_1 = k \cdot 10\exp(\lg\eta_1 - \lg\eta)\dot{\gamma}^{n-1} \end{cases} \tag{6}$$

This method just uses a hypothesis of a same power-law index to obtain unknown fitting equations, and it contributes to understand more information of rheological behavior through a small quantity of experimental data.

4 Conclusions

Compression experiments have provided a method to study the rheological behavior of semi-solid alloys at high solid content, through which some variations of apparent viscosity with variables are obtained. The apparent viscosity increases with decreasing the solid content and shear rate. Another very important factor is the grain size. When the solid content is high, the vicosity increases with decreasing the grain size at high strain rate and decreases with decreasing the grain size at low shear rate. Several fitting equations are obtained through a series of experimental data, and unknown fitting equations also can be obtained through one given apparent viscosity by using the method of time-temperature superposition with a hypothesis of a same power-law index.

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