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Structural evolution of LC_4 alloy in making thixotropic billet by SIMA method①

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[Abstract] The effect of SIMA process parameters on LC_4 alloy's microstructure and the microstructural evolution of various soaking times have been studied. The results show that effective strain in cold deformation before reheating has a great influence on microstructural evolution. Grain size decreases and its shape also approaches to sphericity with increasing effective strain. The amount of liquid phase increases at grain boundaries and grain shape becomes smooth with increasing heating temperature. The main mechanism of grain coarsening is coalescence when eutectic liquid is rare and not totally distributed at all boundaries. Otherwise the main mechanism of grain coarsening is Ostwald ripening and the connection coarsening is more difficult to perform when the regions are nearly full of eutectic liquid.

[Key words] SIMA process; LC_4 alloy; microstructure; coarsening mechanism
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1 INTRODUCTION

Compared with conventional casting and forging technologies, semi-solid metal forming is now becoming a new generation metal forming process due to its easiness to realize near net shape forming, prolonged mold life as well as reductions of shrinkage, porosity and segregation in solidification. Semi-solid metal forming is composed of three main processes: semi-solid materials production, partial remelting and thixoforming. Among these, the semi-solid materials production with uniform and globular microstructure, which can behave as thixotropic characteristics after proper partial remelting treatment, becomes the basis and key to the whole process[1~5]. Strain induced melt activated (SIMA) method has been used to make thixotropic billet. The advantages of SIMA are its simplicity in the process, the uniform and non-dendritic microstructure, no contamination in billet and no using of special equipment. The disadvantage of the process is that the maximum size of billet is restricted by the need to impart the necessary level of deformation in the billet before reheating[6~9]. In this method, cast materials are extruded at elevated temperature to fragment dendritic structure and then cold-deformed above a critical strain to increase the dislocation density in order to obtain fine grains after recrystallization. During heating the cold-deformed alloy to the semi-solid temperature range, recrystallization starts at first with the formation of subgrains and grain boundaries, then melting occurs at the grain boundaries due to the grain boundary's segregation of low melting point elements, and near spherical solid grains are formed and surrounded by low melting eutectic liquid. The size of these grains depends on[7]: 1) alloy chemical composition, which determined the solidus-liquidus temperature interval; 2) microstructure before reheating; 3) heating rate below the solidus, and 4) holding time in the semi-liquid state. The aim of the paper is to obtain near globular microstructure and to evaluate the effect of SIMA process parameters on its resulting microstructure.

2 EXPERIMENTAL

2.1 Raw materials

LC_4 alloy mainly composed of Al, Zn and Mg was employed as experimental material. The exact melting temperature region of the alloy was determined using differential thermal analysis (DTA). The result show that the melting of the alloy starts at 586 ℃ and ends at 626.6 ℃.

2.2 Semi-solid billet making process

The experimental material was hot extruded and treated by a solid solution and natural aging. Then a strain-relief annealing was performed in an annealing condition of heating to 390 ℃, holding for 4 h, and furnace cooling to 150 ℃ and following air cooling to room temperature. Subsequently, cold upsetting was performed at a ram velocity of 1 mm/min using an 1 MN hydraulic press of Tokyo Testing Machine.

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The initial dimensions of specimen for cold upsetting in the SIMA process are 15 mm in diameter and 15 mm in height. The graphite powder was sprayed on two ends of the specimen to reduce non-uniform deformation. Effective strains (the value changed in height/the height of specimen after deformed) are 0.15, 0.28, 0.44, 0.64, 0.84 and 1, respectively. In the end, cold upsetting specimens were heated in electric resistance furnace to a temperature above the solidus point. The specimen's temperature was monitored by a thermocouple and the temperature fluctuation was controlled in the range of ±2 ℃. After that, the specimen was quickly quenched in cool water to fix its transient microstructure.

2.3 Observation and detection of microstructures

Samples were cut at the center part of the quenched specimens cross-sections so as to avoid grain-size difference caused by inhomogeneous compressive deformation. The samples were rough ground, fine ground and polished, then etched with mixing solution of HF, HCL and HNO_3, and their structures were observed under an optical microscope with a magnification of 100 times. The mean size of globular grain was estimated using the Jeffries planimetric method.

3 RESULTS AND DISCUSSION

3.1 Microstructural evolution

Fig. 1 shows initial microstructure of experimental material. Fig. 2 presents the microstructural evolution with various effective strains and heating temperatures. Figs. 2(a), (b), (c) and (d) are microstructures when effective strain reaches 0.15, 0.44, 0.64 and 0.84, respectively, heating at

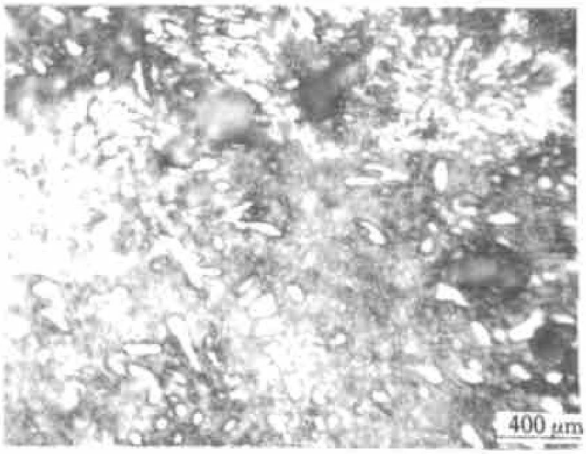

Fig. 1 Initial microstructure of experimental material

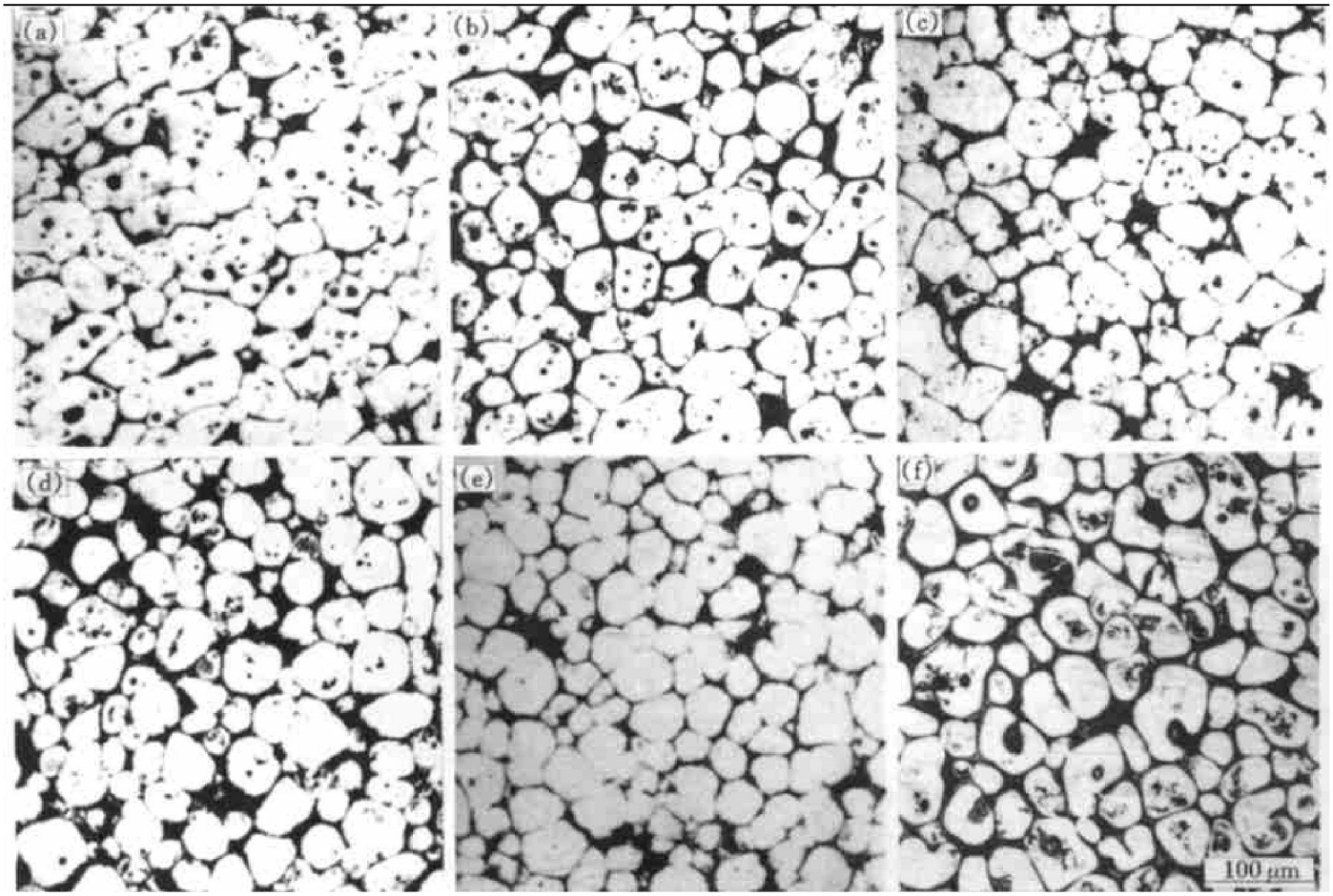

Fig. 2 Microstructure of LC_4 alloy for upsetting in SIMA process
(a) —ε= 0.15, t= 590 ℃; (b) —ε= 0.44, t= 590 ℃; (c) —ε= 0.64, t= 590 ℃;
(d) —ε= 0.84, t= 590 ℃; (e) —Vertical section; (f) —ε= 0.44, t= 600 ℃

590 ℃ for 10 min; Fig. 2(e) shows microstructure of vertical-section corresponding to Fig. 2(d); Fig. 2(f) shows microstructure when effective strain reaches 0.44, heating at 600 ℃ for 10 min. It can be seen from Figs. 2(a), (b), (c) and (d) that grain size becomes smaller and grain shape also approaches globularity with increasing effective strain. Relation of average grain size vs effective strain is shown in Fig. 3. The degree of globular configuration of grain increases obviously when effective strain reaches 0.44. Microstructures of cross-section and vertical-section of samples are all globular grains uniformly refined. In addition, for the same heating temperature and soaking time, although their deformation degrees are different, the amounts of liquid phase of various samples are basically the same, only the liquid phase distributions are different, such as the liquid phase entrapped within grains obviously decreases after effective strain of 0.64. It can be concluded from Figs. 2(b) and (f) that the amount of liquid phase increases and grain shape becomes smoother and grain size decreases slightly with increasing heating temperature at the same effective strain.

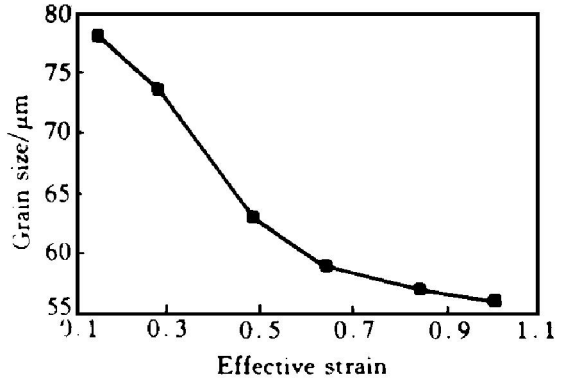

Fig. 3 Average size of grain vs effective strain for upsetting in SIMA process

3.2 Dynamic coarsening process

Fig. 4 shows the microstructures variety with various soaking times (5, 20, and 30 min) after heating at 590 ℃ and deforming at $\varepsilon = 0.44$. It can be seen clearly from Fig. 4 that eutectic liquid is not distributed between the whole primary grains at the initial stage of soaking. The main mechanism of grain coarsening is coalescence, that is to say, several solid grains congregate together and form a new large grain. Eutectic liquid both inside intrinsic grains and between grains are all entrapped within new grains to form liquid pool due to coalescence, which leads to increasing eutectic liquid within the grains. When the soaking time is long enough, the eutectic liquid nearly fills out all grain boundaries and the main mechanism of grain coarsening is Ostwald ripening instead of coalescence. That is to say, small solute grain remelting is deposited on big grains, which leads to the growing of big grains. In the meantime, eutectic liquid entrapped within solid grain decreases and grain shape also shifts to sphericity, which leads to decreasing surface energy. According to LSW theory[11, 12], the diffusion between grains is assumed volume diffusion, and the dynamic equation of grain coarsening during isothermal process is

$$d^3(t) = d^3(0) + kt$$

where $d(t)$ and $d(0)$ are the current and the initial values of the microstructural length scale represented by the average grain diameter, t is the soaking time, 3 is the coarsening exponent and k is constant of coarsening rate.

Relationship between cubic of equivalent diameter of primary grains obtained in this investigation and soaking time is shown in Fig. 5. The results show

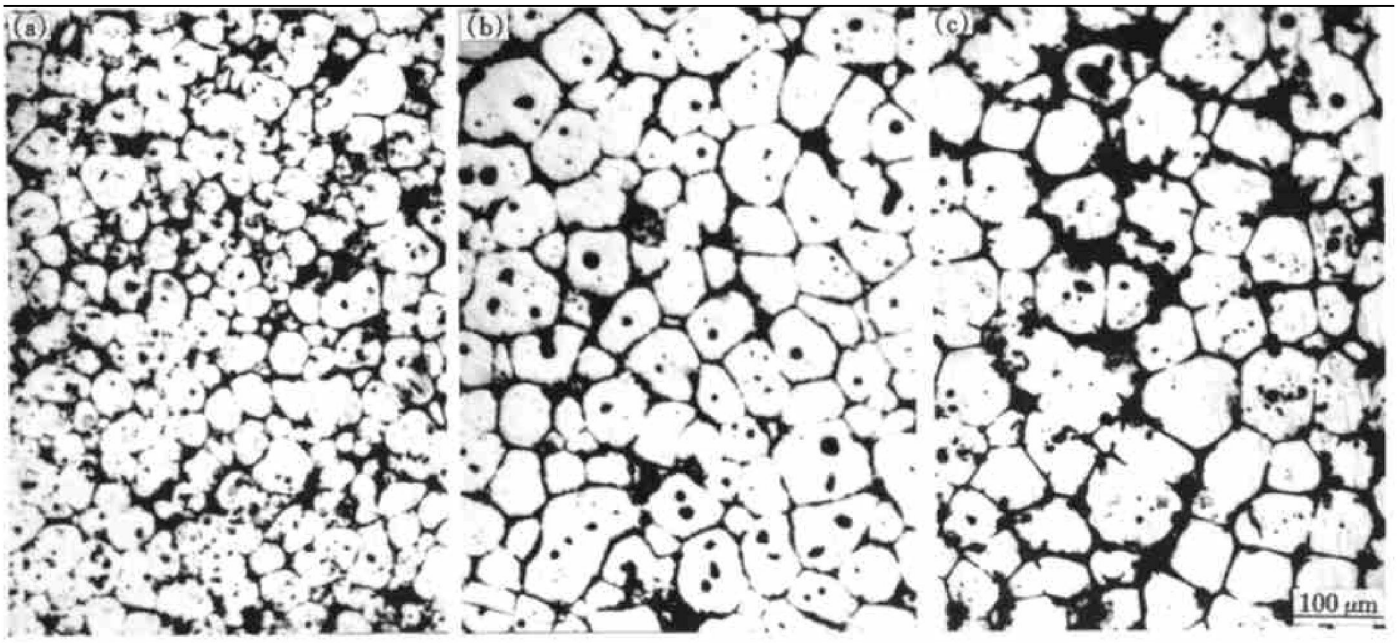

Fig. 4 Microstructural evolution for various soaking times
(a) —5 min; (b) —20 min; (c) —30 min

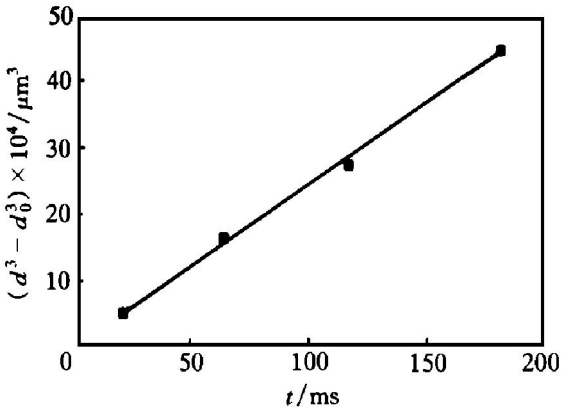

Fig. 5 Relationship between cubic of equivalent diameter of primary grains and soaking time

that the constant of coarsening rate obtained is 2 500 $\mu m^3/s$ and the correlation coefficient is 0.998 7. If the mechanism of grain coarsening mainly belongs to long-range diffusion of grain boundaries and coarsening at the interface, the coarsening exponents are 4 and 2 respectively, and their corresponding correlation coefficients are 0.994 6 and 0.992 8 respectively. The results show that the diffusion between grains more approaches to volume diffusion.

4 CONCLUSIONS

1) With increasing effective strain, grain size becomes smaller, its shape more approaches sphericity and entrapped eutectic liquid within grains decreases obviously for the same heating temperature and soaking times.

2) With elevating heating temperature, liquid phase at grain boundaries increases and grain shape becomes smoother and grain size decreases slightly.

3) At the initial stage of soaking, eutectic liquid is not distributed between the whole primary grains, so the main mechanism of grain coarsening is coalescence. When soaking time is long enough that eutectic liquid nearly fills out all grain boundaries, the main mechanism of grain coarsening is Ostwald ripening instead of coalescence. Moreover the mechanism of grain coarsening belongs to volume diffusion during isothermal process and is satisfied with dynamic equation: $d^3(t) = d^3(0) + kt$.

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