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Rapid solidification of Al-18 %Si hypereutectic alloy in drop tube①

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[Abstract] The drop tube technique was performed to achieve rapid solidification of undercooled Al-18 %Si hypereutectic alloy. The droplets ranging from 60 ~ 1 000 μm in diameter were obtained. The regular polygonal primary Si and lamellar eutectic homogeneously distribute on α(Al) matrix in the droplets larger than 500 μm. While in the droplets smaller than 500 μm the five-star primary Si was found, which is often accompanied by some spherical eutectic grains. The different morphologies of primary Si are due to varied undercoolings. Scanning electron microscopy suggests that the spherical eutectic grain is composed of anomalous eutectic in its core and lamellar eutectic radiating outside from its periphery. Such eutectic microstructure is presumed to be the result of combining large undercooling, microgravity with containerless processing during free fall.

[Key words] rapid solidification; undercooling; Al-Si alloy; drop tube

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1 INTRODUCTION

Research on solidification in drop tube processed droplets has aroused extensive interest of metallurgists for three main reasons: (1) falling droplets are effectively in a microgravity state[1], (2) contamination is avoided during solidification and high-purity materials can be produced, (3) containerless processing which suppresses heterogeneous nucleation of melts and fast cooling with inert gas can achieve high undercooling prior to nucleation, which makes rapid solidification possible[1]. Both microgravity and high undercooling are of great importance in fundamental studies of solidification processes and the resultant microstructures.

Because of its low expansion coefficient and excellent wearability, Al-Si hypereutectic alloy has been widely used as casting pistons. However, the coarse primary Si greatly decreases the strength of the alloy and restricts its industrial applications. Therefore the modification of coarse primary Si is necessary. The modification can be achieved by three kinds of techniques: ultrasonic treatment[2], chemical addition (for example Cu-P binary master alloy and Al-Cu-P ternary master alloy) and increasing cooling rates. They can both transform the morphology of primary Si from coarse irregular flakes to finer regular polygonal Si.

The purpose of the present work is to explore the formation mechanism of different morphologies of primary Si and spherical eutectic grains. The calculation of the cooling rate during free fall is also carried out to prove that rapid solidification can be achieved in drop tube.

2 EXPERIMENTAL

The experiments were performed in a 3 m drop tube. The samples of Al-18 %Si hypereutectic alloy were prepared from pure Al (99.99 %) and Si (99.99 %) and each has a mass of about 0.5 g. At the beginning of experiment, a sample was placed in a quartz tube with a *d* 0.3 mm orifice at the bottom. The quartz tube was installed at the top of the drop tube. The drop tube was then evacuated to 2×10^{-4} Pa and backfilled with the mixture of helium and argon gases to about 0.1 MPa. The sample was inductively melted at a certain temperature above its melting point and dispersed into many droplets by jetting through the orifice with exerting a differential pressure on the melt. Droplets ranging from 60 ~ 1 000 μm in diameter were collected at the bottom of the drop tube and processed according to standard metallographic procedures. The microstructures were analyzed with an XJG-05 optical microscope and a JXA-840 scanning electron microscope.

3 RESULTS AND DISCUSSION

The typical microstructures of different droplets are shown in Fig.1. In the droplets larger than 500 μm the microstructure illustrated in Fig.1(a) is commonly observed. The regular polygonal primary Si and plate-like eutectic Si distribute homogeneously

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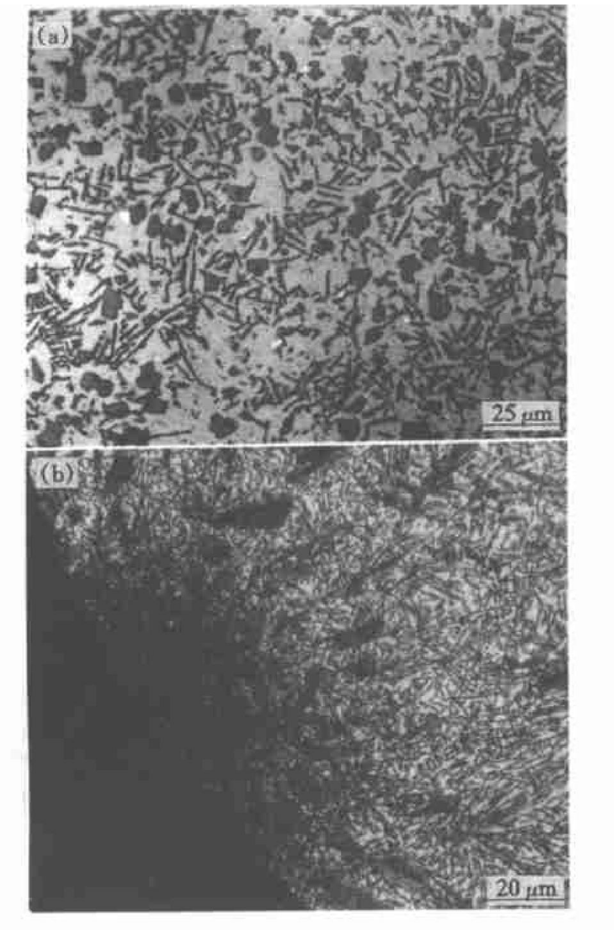

Fig.1 Microstructures observed in different droplets
(a) —Regular polygonal primary Si + lamellar eutectic;
(b) —Five-star primary Si + eutectic grains + lamellar eutectic

on eutectic α(Al) matrix. Such microstructure is similar to the modification microstructure of Al-Si hypereutectic alloy with 0.015 %P addition. While it is found that the five-star primary Si is accompanied with some eutectic grains (shown in Fig.1(b) and Fig.2) at the periphery of the droplets smaller than 500 μm. SEM image indicates that such eutectic structure is composed of anomalous eutectic in its core and lamellar eutectic radiating outside from its periphery, which is illustrated in Fig.3.

According to Newtonian cooling model[3,4], calculation of the cooling rate versus droplet diameter is accomplished and the result is presented in Fig.4. The cooling rates of the droplets with their size at the two extremes in the present work are 3.8×10^5 K/s and 3.9×10^3 K/s, respectively. At so high cooling rate, rapid solidification can be achieved. Obviously, the cooling rate increases when the droplet diameter decreases. Meanwhile, the melt subdivision makes the smaller droplets contain fewer heterogeneous nuclei than the larger ones. These two factors makes the smaller droplets achieve relatively large undercooling.

So it can be presumed that the different morphologies of primary Si are due to varied undercoolings. The five-star primary Si forms at larger undercoolings. There are many fields rich in Si in the alloy melt, which makes it easy the formation of primary Si nucleus with lowest interfacial energy on ten close-packed {111} faces because of larger driving force. The Si nucleus is composed of five twinned tetrahedrons, which makes it possible that the advancing twin growth front is fivefold[5]. So the five-star pri-

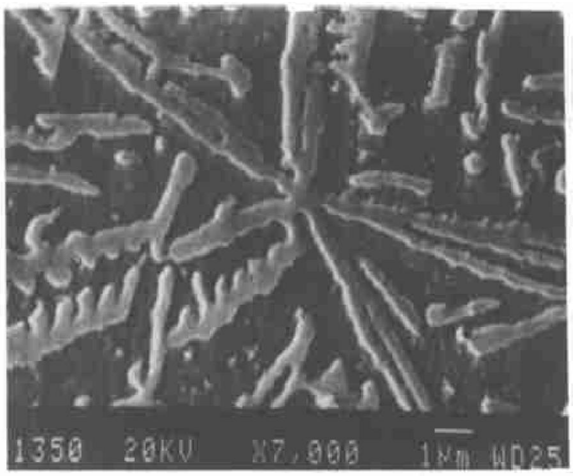

Fig.2 SEM image of five-star primary Si

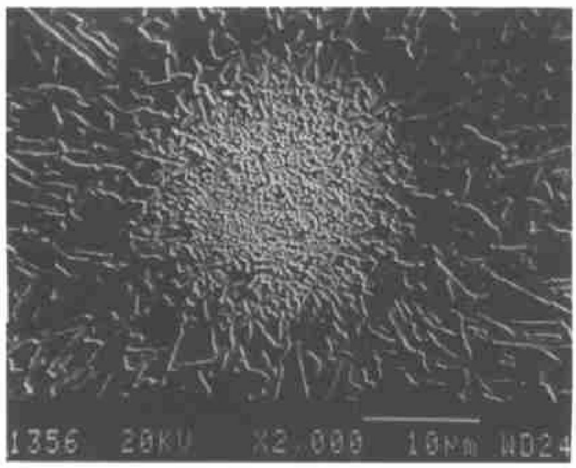

Fig.3 SEM image of eutectic grains

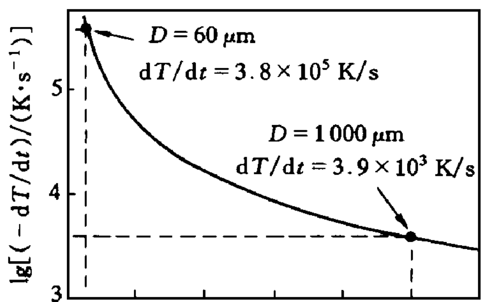

Fig.4 Cooling rate dT/dt versus droplet diameter D

mary Si forms. In contrast, the microstructure illustrated in Fig.1(a) forms at relatively small undercoolings. TPRE (Twin plane re-entrant) growth is suppressed and the primary Si grows into octahedrons in the ⟨100⟩ direction. Therefore its sectional view is regularly polygonal.

The spherical eutectic grain has not been reported in the previous references about Al-Si hypereutectic alloy. While this kind of microstructure is often observed in the eutectic system alloys processed in drop tube[6~8]. So it is reasonable to presume that such microstructure results from some specific effects provided by drop tube processing. On one hand, at the periphery of a falling droplet large undercooling can be achieved more easily because of rapid quenching of inert gases during free fall. So copious isolated nucleation will take place preferentially at the periphery and the anomalous eutectic forms. Subsequently for the release of large amount of latent heat, the temperature at the advancing growth front increases and lamellar eutectic forms at smaller undercoolings. Trivedi et al[9] presented that the temperature-dependent diffusion coefficient decreases significantly at large undercoolings. The diffusion distances for isolated eutectic phases becomes smaller than half of the interlamellar spacing so that no advantage is obtained for the efficient solution distribution by the cooperative growth. Therefore the cooperative growth is replaced by copious isolated nucleation and rapid growth, which leads to the formation of anomalous eutectic. While at small undercoolings the cooperative growth is easier and lamellar eutectic forms. WEI[10,11] proved experimentally that there is a "lamellar eutectic-anomalous eutectic" transition with the increase of undercooling. On the other hand, drop tube processing can make the falling droplet be in a microgravity and containerless state, which makes the heat and mass transfer during solidification be spatially symmetric. So the spherical eutectic grains form. Summarily, such microstructure results from combining large undercooling, microgravity and containerless processing.

4 CONCLUSIONS

1) Al-18 %Si hypereutectic alloy is processed in a 3 m drop tube and the droplets ranging from 60 ~ 1 000 μm in diameter are obtained. The calculation of cooling rate versus droplet diameter is also carried out. The cooling rates of the extreme-sized droplets are 3.8×10^5 K/s and 3.9×10^3 K/s, respectively, which makes rapid solidification possible.

2) The regular polygonal primary Si and five-star primary Si are respectively observed in the different-sized droplets. The variance is due to different undercoolings.

3) The spherical eutectic grain results from combining large undercooling, microgravity and containerless processing.

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