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Formation and growth of solid particles in shear flow

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Formation and growth of solid particles of Sn–15% Pb and Al–40% Zn were studied using a Couette type viscometer in isothermally and continuously cooled shear flows. Solid particles come mainly from broken dendrites in the initial stage of solidification. With increase of solid fraction, solids grow in an equiaxial and merging way in the flow of the partially solidified alloy. Mergence happens between two particles of small misorientation, and merging growth is performed by the growth of protruding parts on the solid–liquid interfaces of the solids.

1. Introduction

Partially solidified alloys which contain solid particles suspended in the liquid are obtained in flow when alloys are undergoing solidification. They have wide prospective applications in die-casting of high melting temperature alloys, forming metal matrix composites, etc. [1, 2]. In recent years, more attention has been paid to knowing how an alloy solidifies in flows [3, 4], but opinions vary as to where the solid particles appear and how they grow in different stages of solidification. In this paper, the formation and growth of solid particles of solidifying alloys in shear flow were studied in order to know better and to control the microstructures of the alloys in use.

2. Experimental procedure

Hypoeutectic alloys, Sn–15% Pb and Al–40% Zn, are prepared using pure tin, lead, aluminium and zinc. Alloys are solidified in the gap between cylinders of the Couette type of viscometer which can sample the alloy by quenching within one second. As the inner cylinder rotates, shear flow is generated in the melted alloys. Solid fraction of the alloy (φ_s) is calculated by Scheil equation [5], shear rate of the alloy is estimated by the following formula [5, 6]:

$$\dot{\gamma} = \frac{2\omega}{1 - (R_1/R_2)^2} \tag{1}$$

Here, ω is the angular velocity of the inner cylinder, R_1 and R_2 the radii of the inner and outer cylinders, respectively. ($R_1 = 3.50$ mm, $R_2 = 4.00$ mm). In the experiment, isothermal cooling with cooling rate less than $0.05°\,C\,min^{-1}$ and continuous cooling with cooling rate $0.45°\,C\,min^{-1}$ were used. The microstructures of partially solidified alloys are examined by scanning electron microscope (SEM: X-650) and transmission electron microscope (TEM: 2000EX). The size distribution of solid particles is measured according to the greatest length of separate solids on polished surfaces of two specimens with a total field of vision more than $50\,mm^2$ under each condition.

3. Results and analyses

3.1. Formation of solid particles

When the solid fraction is less than 0.1, well developed dendrites are found in partially solidified alloys with the size about 200 μm. They come mainly from the wall of the outer cylinder, and their growth and break into slurries are thought to be concerned with the existence of the fluid boundary layer which is of the same order of size as the dendrites. Dendrites in the slurry flow suffer a force which is directly proportional to shear rate and solid fraction, so that they are gradually broken into pieces and become more particle-like with increase of shear rate and solid fraction. Dendrites are seldom seen when the solid fraction is greater than 0.3 for Sn–15% Pb and 0.4 for Al–40% Zn. Thus, solid particles of partially solidified alloys are not all directly from dendrites, they are from the growth of broken dendrites in the initial stage of solidification, especially when the solid fraction is high. Figs 1 (a) and (b) show the morphology of solids of partially solidified Sn–15% Pb and Al–40% Zn in continuous cooling, the solid fraction being about 0.22. It can be seen from Fig. 1 that most of the solids are spheroidal in shape.

3.2. Growth of solid particles

As dendrites are broken into pieces, the solids grow in the flow of slurries. Two growth mechanisms are found: equiaxial growth and merging growth. Fig. 2 gives the size distributions of solid particles of Sn–15% Pb in continuous cooling with shear rates of 50(1/S) and 200 (1/S). The peaks of the size distribution curves move to the right due to equiaxial growth of solids, and they broaden due to merging growth. The higher the solid fraction, the more important role the merging growth plays in alloy solidification in flows. Compared with the flow in shear rate 200(1/S), the size distribution curve of the flow in shear rate 50(1/S) tends to be gentle with lower solid fraction. Therefore, mergence will be obstructed by high shear rate flows. Fig. 3 shows the average size of solids against solid fraction of Sn–15% Pb under

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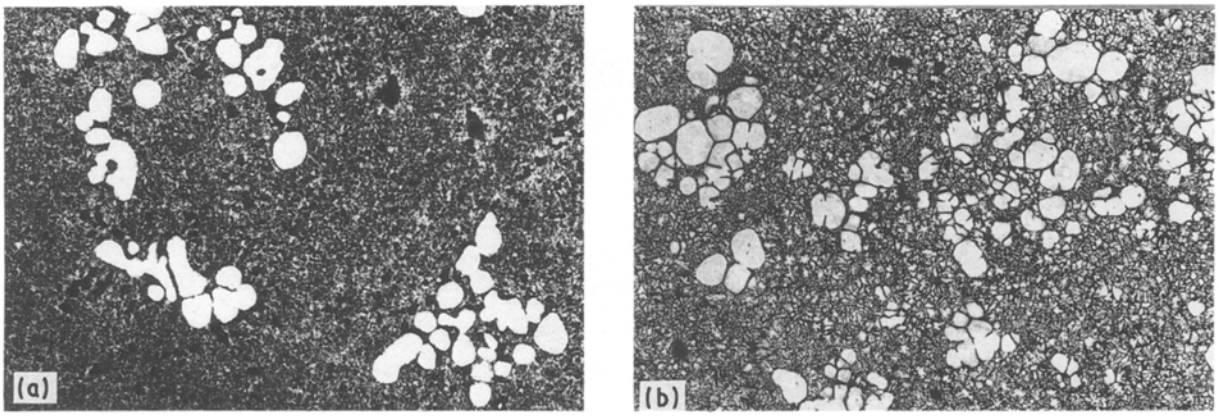

Figure 1 Morphology of solids of partially solidified alloys in continuous cooling with solid fraction 0.22 and shear rate 100 (1/s). (a) Sn–15% Pb; (b) Al–40% Zn. (× 100)

isothermal and continuous cooling conditions. With the increase of solid fraction, solid size increases more quickly in continuous cooling than in isothermal cooling, even though the solid size at low solid fraction is greater in isothermal cooling than in continuous cooling. This is attributed to quicker merging growth under the continuous cooling condition when solid fraction increases.

3.3. Characteristics of mergence

The solid particles are not homogeneously distributed in the slurries in shear flow, but agglomerated. There are liquid films between solids in agglomerates, so that they will either leave the agglomerates or gather again in the flow. Mergence happens between gathered particles. Fig. 4 shows the merged solids of Sn–15% Pb in isothermal cooling with shear rate 200(1/S). It can be seen there that most of the merged solid particles come from two-particle merging. Through analysing the misorientation of merging solids by locating the Kikuchi pattern poles [7], we found that most merging solids have small misorientations. Fig. 5 shows the misorientation analysis for Al–40% Zn. The results show that interficial energy comes into action in solid-merging [8], even when solids are moving in highly sheared flows of liquid. Besides, when two particles are close in favourable orientation, they will not merge if they are separated by liquid flow if there is poor attraction between the two merging particles. Fig. 6 shows two merging solids of Sn–15% Pb. It can be seen that mergence is performed by the growth of protruding parts on the solid–liquid interfaces of the solid, which are the sign of unstable growth of solid particles for the negative temperature gradient in the liquid near the solids. That is why the mergence of solids is accelerated by increase of cooling rate and obstructed by increase of shear rate.

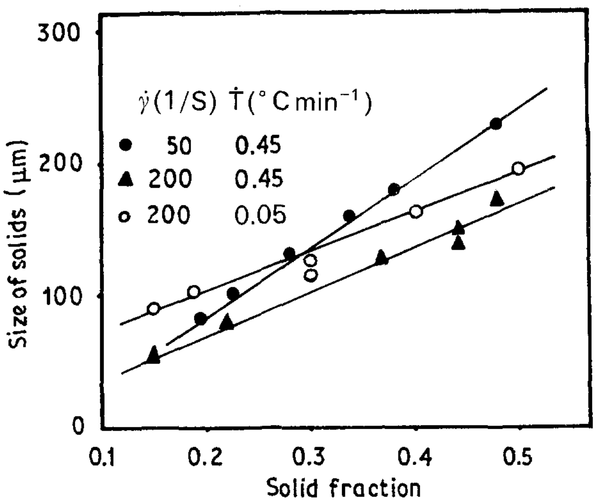

Figure 3 The average solid size of Sn–15 Pb against solid fraction under isothermal and continuous coling conditions. $\dot{T}$ stands for the cooling rate.

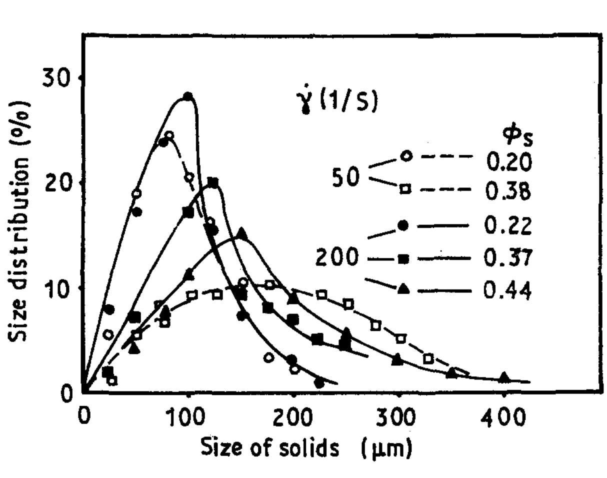

Figure 2 Size distributions of solid particles of Sn–15% Pb in solidification processing in the continuously cooled shear flow with shear rate 50(1/S) and 200(1/S).

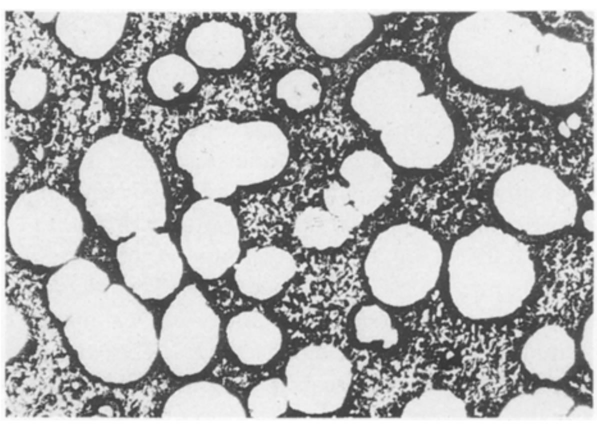

Figure 4 Solid particles of Sn–15% Pb are merging in the flow of slurry with shear rate 200 (1/S) in isothermal cooling. Solid fraction is 0.51. (× 50)

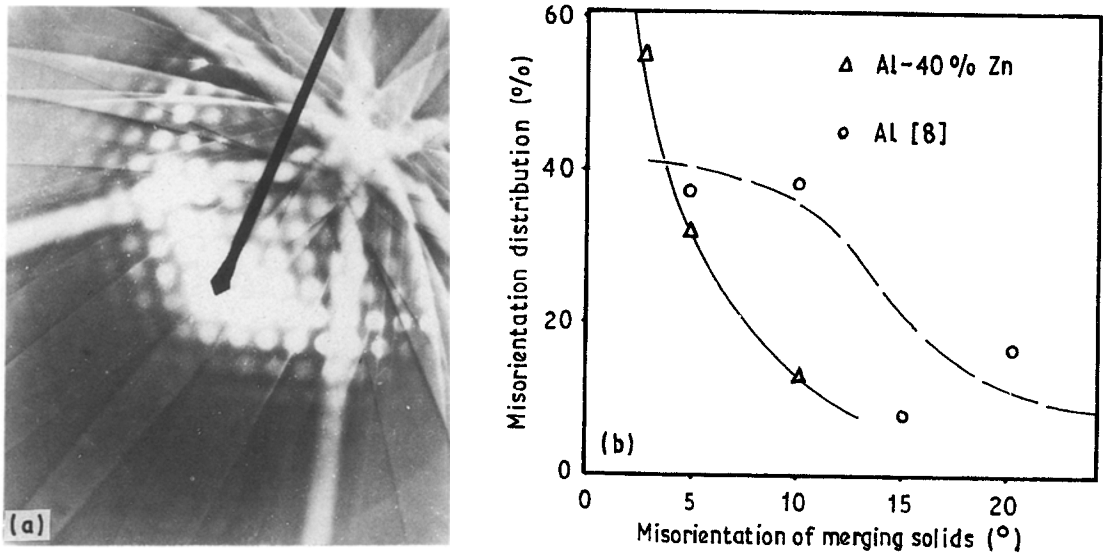

Figure 5 The orientation analysing results of Al–40% Zn in isothermal cooling with shear rate 200 (1/S) and solid fraction 0.51. (a) The Kikuchi pattern of a solid in merging (TEM). Camera length is 30 cm. (b) The distribution of misorientations (α) derived from 31 pairs of merging solids. Results marked by Δ are sectioned into 3 : $\alpha \leqslant 3°$, $3° < \alpha \leqslant 6°$, $\alpha > 6°$; results marked by 0 are sectioned into 4 : $\alpha \leqslant 5°$, $5° < \alpha \leqslant 10°$, $10° < \alpha \leqslant 15°$, $\alpha > 15°$.

4. Discussion

Many observations and results in the paper support the clustering model of thixotropy for partially solidified alloys suggested by Joly and Mehrabian [7–9]. However, it is also found that some boundaries of merging particles are formed of dislocations without solute enrichment, which is evidence in favour of the model of plastic bending suggested by Vogel [10]. But it needs confirmation that the boundaries without solute enrichment do come from plastic bending, because there is quick solute diffusion in solids at high temperatures. From our investigations of solidification of alloys in shear flow by successive sampling through quenching, it is certain that mergence of solids is one of the important mechanisms in solid growth in flow.

5. Conclusion

The particle-like solids in partially solidified Sn–15% Pb and Al–40% Zn in shear flow come mainly from the fragments of dendrites at the beginning of solidification. There are two mechanisms in solid growth, and when the solid fraction exceeds 0.3, the later mechanism plays the main role. The merging growth is accelerated by increase of cooling rate and obstructed by increase of shear rate. The mergence is performed by the growth of extruding parts on solids' surfaces, and there are small misorientations between merging solids.

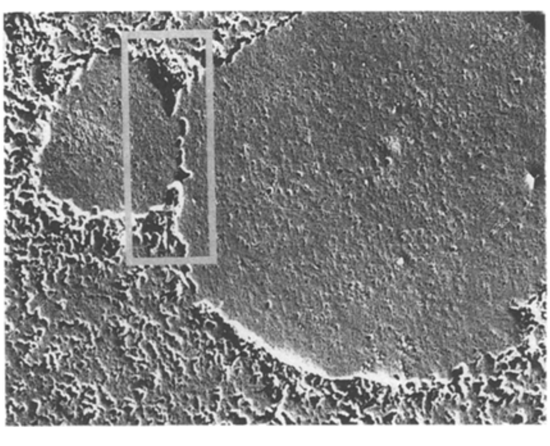

Figure 6 Two solid particles of Sn–15% Pb are merging in the slurry flow by growth of extruding parts on each particle surface under continuous cooling conditions (SEM). Shear rate is 100 (1/S) and solid fraction is 0.34. ($\times$ 110)

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