Solidification microstructures in a short fiber reinforced alloy composite containing different fiber fractions

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Abstract: The solidification microstructures and micro-segregation of a fiber reinforced Al-9 Cu alloy, containing different volume fractions of Al_2O_3 short fibers about 6 μm diameter and made by squeeze casting have been studied. The results indicate that as volume fraction of fiber V_f increases, the size of final grains becomes finer in the matrix. If $\lambda_f/\lambda > 1$, the fibers have almost no influence on the solidification behavior of the matrix, so the final grains grow coarse, where λ_f is the average inter-fiber spacing and λ is the secondary dendrite arm spacing. While if $\lambda_f/\lambda < 1$, the growth of crystals in the matrix is affected significantly by the fibers and the grain size is reduced to the value of the inter-fiber spacing. The fibers influence the average length of a solidification volume element L of the matrix and also influence the solidification time θ of the matrix. As a result of fibers influencing L and θ, the micro-segregation in the matrix is improved when the composite contains more fibers, although the level of the improvement is slight. The Clyne-Kurz model can be used to semi-quantitatively analyze the relationship between V_f and the volume fraction f_e of the micro-segregation eutectic structure.

Key words: short fiber; composite; fiber fractions; volume fraction; micro-segregation

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Al_2O_3 short fiber reinforced aluminium alloy has a lot of superior properties, including low weight, low thermal expansion coefficient, high strength-to-weight and stiffness-to-weight ratio, high-temperature resistance, high corrosion resistance and so on. All these excellent properties promise the material a good application perspective in the aerospace automotive, and mechanical engineering areas. Despite the widespread application of the material, however, there have, as yet, been few reports available on the solidification of it. In this study, Al_2O_3 short fibers were chosen as the reinforcement, Al-9Cu was chosen as the matrix alloy, and the microstructures and micro-segregation in composites containing different fiber fractions were analyzed.

1 Experimental Procedure

The fiber reinforced metal matrix composites used in the present study was initially prepared by squeeze casting. The matrix alloy was Al-9.0 wt %Cu and was prepared using super purity aluminium (99.99 % Al) and high purity copper (99.9 % Cu). The related parameters about the fibers are shown in Table 1.

In the first procedure, fiber preforms were prepared by compressing the appropriate weight of fibers, according to the need for different volume fractions of fiber in the final composite, into a cylindrical steel mould of 51.5 mm in diameter and 30 mm in length. After being dried and pre-heated, one preform was put into the pre-heated squeeze casting mould, then molten Al-9Cu alloy liquid was poured in, which infiltrated into the fiber preform under an external pressure of about 10 MPa. The fiber volume fractions V_f of fiber preforms used in the composite bars were such as to give 0.1, 0.15, and 0.2 respectively. These specific values of V_f were measured by the quantitative analysis system with a CS AXLOSKOP2 microscope. These bars were then cut into shorter lengths to provide samples for remelting, which were 15 mm in diameter and 12 mm in height. A small hole, 2 mm in diameter, was drilled along the central axis of each short cylindrical sample, to a depth of 7.5 mm. This was to accommodate a thermocouple during the next experimental procedure.

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Table 1 Chemical composition and physical parameters of Al_2O_3 short fibers

Composition		Physical properties				
Al_2O_3 wt%	SiO_2 wt%	Diameter μm	Density g/cm³	UTS MPa	E GPa	Primary phases
80	20	6-8	3.14	600	240	α - Al_2O_3, $3Al_2O_3$-$2SiO_2$

Some other technological parameters in the squeeze casting process were as follow: pre-heated temperature of mould was 350 ℃, preform was at 450 ℃, molten alloy was at 800 ℃ and external pressure maintaining time was 1 min.

During the next procedure, the shorter composite bars containing different fiber volume fractions were remelted and resolidified under controlled conditions in order to study the influence of the change of V_f on the microstructures in the composites. The remelting temperature was 720 ℃. The remelting arrangement was as shown in Fig.1. The furnace shown consisted of four main parts: (1) an outer alumina furnace; (2) a cooling tank containing air; (3) a graphite cylinder with three 17 mm diameter holes drilled parallel to the axis and spaced at 120 degree angles around the section of the cylinder; and (4) a central steel rod, 2 mm in diameter, attached to a segment plate beneath the graphite cylinder. Three composite samples containing different V_f were placed as shown and their temperatures were measured with chromel-alumel thermocouples made from 0.2 mm diameter wire. The samples were heated to 720 ℃, allowed to equilibrate for about half an hour prior to being cooled in the fully liquid state. The rod attached to the segment plate was then rotated through an appropriate angle, and the three samples fell from the graphite cylinder to the cooling tank. Traces of temperature against time were recorded by a chart recorder, and 20 mV of the thermocouple output was backed off to improve the resolution of the temperature measurements. Temperature resolution was thus approximately ± 0.5 K. The total solidification time θ_t of each sample was determined by the temperature shown on thermocouple and the trace of temperature against time was recorded by the chart recorder. After being polished and etched, the three group samples were observed on XL-30 SEM and measured using a CS AXLOSKOP2 metallographic microscope.

2 Results and Discussion

Micrographs of the remelted samples containing different V_f are shown in Fig. 2. From these it can be seen that the average size of grains in the matrices becomes finer as V_f rises. The real average sizes of grains measured according to GB6394 are as follow: d_1= 48 μm, d_2= 36 μm, d_3= 23 μm. In Fig. 2 (a) the value of the fiber fraction V_{f1} was the lowest, so the average inter-fiber spacing λ_{f1} was the longest, which can be calculated using the equation $\lambda_{f1} = (1-V_f)\, d_f/V_f$ [1], where d_f is the diameter of the fibers. λ_{f1} is worked out to be 54 μm. Since λ_{f1} is the lowest, the growth of matrix crystals in sample 1, shown in Fig.2 (a), was the least affected by fibers. This means crystals in the matrix can grow quite freely. If there had been no fibers in sample 1, the secondary dendrite arm spacing λ_a in an un-reinforced alloy under the same solidification conditions could be calculated according to the equation $\lambda = 7.5 \times 10^{-6} \theta_t^{0.39}$ (m) [2], where θ_t is total solidification time. For sample 1, θ_t has been measured to be 103 s through the arrangement shown in Fig.1. λ_a had a value of 45 μm. Obviously, $\lambda_{f1} > \lambda_a$. Thus the secondary dendrite arm spacing λ_1 in the matrix of sample 1 can be set equal to λ_a according to the study of Mortensen et al. [3]

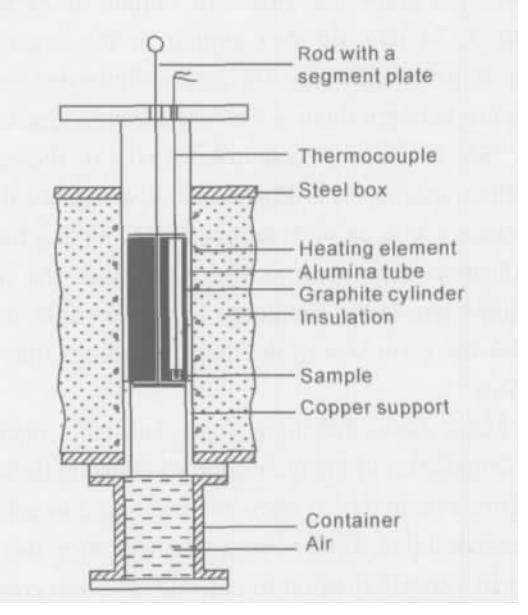

Fig.1 Remelting and resolidifying arrangement

For sample 2, θ_t is determined by experiment as 110 s. Thus the secondary dendrite arm spacing λ_a in an un-reinforced alloy would have a value of 47 μm, while λ_{f2}= 34 μm. It can be seen that $\lambda_{f2} < \lambda_a$. This indicates that, even for a dendrite tip advancing parallel to the fiber direction, the dendrite growth with normal side-branching

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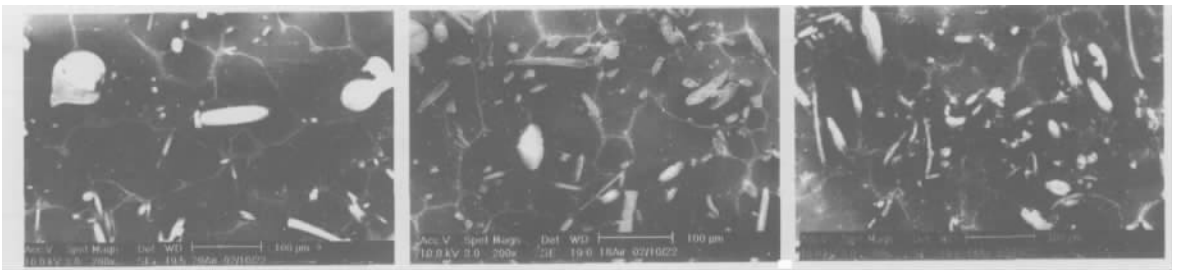

(a) Sample 1, V_f=0.1 (b) Sample 2, V_f=0.15 (c) Sample 3, V_f=0.2

Fig.2 Micrographs of remelted composites with different fiber fractions

will be severely inhibited by the fibers in this sample. The surface of Al_2O_3 short fiber can not serve as a heterogeneous nucleation site for primary phase of the Al-Cu alloy [4-6], and hence the primary α-phase nucleates in the space between the fibers and grows towards the fiber. With Al_2O_3 short fiber having a high heat-accumulating coefficient, liquid around them has a lower cooling rate, that is, the last regions of solute-rich liquid to solidify are located around the fibers. Therefore, the final crystal size of matrix in sample 2 can be approximately recognized as equal to λ_{f2}, or 34 μm. Similarly, for sample 3, λ_{f3}= 24 μm, and λ_c= 50 μm (θ_3= 119 s). The grain size in matrix of the sample 3 can be predicted as 24 μm.

Comparing the two group values, it can be seen that the two inferred grain size values of sample 2, 34 μm and sample 3, 24 μm are very similar to the experimental values 36 μm and 23 μm. The author thinks that the slight difference between them is because of some measurement error. So it can be concluded that, if the average inter-fiber spacing λ_f is less than the secondary dendrite arm spacing λ in an un-reinforced alloy having the same solidification conditions as the composite, the average final grain size in the composite is reduced to be equal to λ_f. Also the grain size in the matrix becomes finer as V_f increases.

Fig. 2 also shows that there is gray substance distributed at the boundaries of the grains and in the neighborhood of the fibers. For an Al-Cu alloy, the last liquid to solidify is copper-rich liquid. Under eutectic temperature, this liquid results in a transformation to eutectic structure composed of α-Al and θ-$CuAl_2$ phases. In order to find out the eutectic fraction in the final solidified composite with different V_f, and to further measure the degree of micro-segregation in the matrix, the Clyne-Kurz modification[7-8] of the Brody-Flemings micro- segregation model which incorporated back-diffusion of solute in the solid can be quoted to have a semi-quantitative analysis. Using such a model, composite profiles can not be predicted, only the eutectic fraction of the final structure is calculated. This parameter is, however, obviously a measure of the degree of homogenization. In the Clyne-Kurz model, the eutectic fraction f_e is given by

$$f_e = 1 - \frac{1}{1-2\alpha' k_o}\left[1-\left(\frac{C_e}{C_o}\right)^{\frac{1-2\alpha' k_o}{k_o-1}}\right] \quad (1)$$

where $$\alpha' = \alpha\left[1-\exp\left(-\frac{1}{\alpha}\right)\right] - \frac{1}{2}\exp\left(-\frac{1}{2\alpha}\right) \quad (2)$$

and $$\alpha = \frac{D_s \theta_t}{L^2} \quad (3)$$

In these equations, k_o is the distribution coefficient (dimensionless), C_e is the eutectic composition (wt%), C_o is the alloy composition (wt%), D_s is the solute diffusivity in the solid (mm^2/s), L is the average length of a solidification volume element (mm), α is diffusion parameter (dimensionless) and α′ is the modified diffusion parameter (dimensionless).

As the fiber fraction in a composite increases the potential heat of crystallization that one unit of material can release is predicted to reduce, which would result in the cooling rate increasing. On the other hand, the heat accumulating coefficient of the composite increases as V_f rises, which would lead to the cooling rate decreasing. So the cooling rate of the composite is determined by these two conflicting factors. For an Al_2O_{3f}/Al-9Cu composite, the fiber-influenced heat accumulating capability of the material is obviously the dominant factor. The same conclusion can be obtained from practical calculation with the relative parameters. Therefore, the cooling rate of the composite decreases with the increasing of fiber fraction, which means solidification time θ becomes longer. Also from above, it can be seen that the increasing fiber fraction reduces crystal size. This can furthermore shorten the average length of a solidification volume element L. It is apparent then that with a larger value of V_f, composite

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solidifies at a shorter length in a longer time. With the help of mathematical knowledge, it is known that f_e is a decreasing function of α. Since α is proportional to θ_f/L, f_e is then a decreasing function of θ_f/L. Thus, with more fibers in the composite, less eutectic is predicted to develop in matrix as is indeed observed in Fig. 2. The specific calculation is as follows:

For sample 1: θ_f= 103 s, L = $d_f/2$ = 24 μm,
then α= 0.081, f_e = 0.189
For sample 2: θ_f= 110 s, L = $\lambda_{f2}/2$ = 17 μm,
then α= 0.187, f_e = 0.177
For sample 3: θ_f= 119 s, L = $\lambda_{f3}/2$ = 12 μm,
then α= 0.402, f_e = 0.164

In order to test the results calculated with the Clyne-Kurz model, a CS AXLOSKOP2 metallographic microscope and the MLAPS (MICRO-IMAGE ANALYSIS PROCESS) software were used in the experiment to measure quantitatively the eutectic fraction in each sample. The results are listed in Table 2.

Table 2 Measured values and calculated values about eutectic volume fraction in each sample

Sample number	1	2	3
Measured value of eutectic fraction	0.191	0.179	0.163
Calculated value of eutectic fraction	0.186	0.177	0.164

It can be seen from the above results that the Clyne-Kurz model can be used to semi-quantitatively calculate the influence of V_f on eutectic volume fraction f_e, and with fiber fraction increasing, the eutectic micro-segregation lessens, although the lessening is quite slight. Even so, it is still very important from the point of view of mechanical properties. It is already known that more brittle phase, which is θ-$CuAl_2$ phase in this work, at the fiber-matrix interface or on the boundaries of matrix grains can adversely affect fracture strength. So in order to obtain optimum mechanical properties, properly more fibers should be added into the composite after solving the interface problems between fibers and matrix.

3 Conclusions

(1) When the composite has a higher value of fiber fraction, the size of the final solidified crystal grains in the matrix can be made finer. Also if $\lambda_f/\lambda > 1$, the crystals grow very freely and coarsely, while if $\lambda_f/\lambda < 1$, the grain size of the matrix is reduced to the value of the average inter-fiber spacing.

(2) As the fiber volume fraction V_f increases, the average length of a solidification volume element L is shortened and the total solidification time θ_f is extended.

(3) The fraction of eutectic micro-segregation, f_e, is reduced as V_f rises, although the level of the reduction is not very obvious.

(4) The influence of fiber volume fraction V_f on eutectic fraction f_e can be semi-quantitatively calculated by using the Clyne-Kurz model.

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