

## ACCELERATING BRIDGMAN SOLIDIFICATION OF Al-3wt%Fe ALLOYS CONTAINING 1wt%Cu, Mg, Si OR 0.1wt%V

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**ABSTRACT** Solidification microstructure of Al-3wt%Fe alloys containing 1wt% of either Cu, Mg or Si or 0.1wt%V was investigated by means of accelerating velocity Bridgman growth over the velocity range 0.03 to 1.0mm/s. Critical velocities for microstructural changes were determined and intermediate transitions which were not found previously by constant velocity Bridgman growth in the alloys were revealed.

**Keywords:** *accelerating Bridgman growth, aluminium alloy, eutectic, solidification*

### 1. INTRODUCTION

During previous studies of intermetallic phase selection in the Al-Fe system [1-6], the constant growth velocity Bridgman technique (CBG) was usually utilised in which samples were unidirectionally solidified at fixed growth velocities. However, since CBG experiments have to be performed with finite velocity intervals while the microstructural changes involved occur over relatively narrow velocity intervals, some transitions can be missed. It was shown by CBG that additions of third alloying elements resulted in significant changes in the solidification microstructure of Al-3wt%Fe [3-6]. The present study described a further investigation of the solidification microstructure of Al-3wt%Fe alloys containing 1wt% of either copper, magnesium or silicon or 0.1wt% vanadium by using an accelerating Bridgman growth (ABG) technique in which samples were grown at continuously increasing growth velocity as solidification proceeded so that the velocity for each structural transition could be determined precisely. Earlier papers [5,6] reported the results of CBG for these alloys. The ABG experiments in the present study were designed to impose an acceleration compatible with the particular growth velocity range to be covered for these alloys, which was of particular interest with respect to the microstructural changes and transitions between the non-equilibrium and metastable eutectics in the Al-Fe system found in previous studies [1-6].

### 2. EXPERIMENTAL

Five alloys, Al-3.0Fe, Al-2.85Fe-0.96Cu, Al-2.84Fe-1.11Mg, Al-2.91Fe-1.08Si and Al-2.85Fe-0.12V (in wt%), were supplied as 10mm diameter rods by Alcan International Ltd based on 99.99%Al and an Al-5.2wt% master alloy. The rods were subsequently swaged and drawn to fit into 2mm ID×200mm long alumina tube for accelerating Bridgman growth in a vertical Bridgman facility over the velocity range 0.03 to 1.0mm/s which was chosen according to the previous results obtained from CBG [5,6]. The acceleration was achieved by using a stepper motor whose rate could be varied (increased or decreased) to drive the sample-withdrawal system in the Bridgman facility used. In practice, the ABG experiments were carried out separately in three sub-ranges 0.03 to 0.33mm/s, 0.33 to 0.67mm/s and 0.67 to 1.0mm/s with accelerations of  $3.61 \times 10^{-4} \text{mm/s}^2$ ,  $1.11 \times 10^{-3} \text{mm/s}^2$  and  $1.86 \times 10^{-3} \text{mm/s}^2$ , respectively. Since the growth velocity  $V$  at which any point along the specimen solidified could be calculated from the initial velocity  $V_0$ , acceleration  $a$  and the distance  $d$  from the

point at which solidification started, a continuous record of microstructure of the solidified specimen as a function of growth velocity  $V$  could be obtained.

Standard polishing techniques were used followed by etching in Keller's reagent for optical observation. With the help of TEM examinations on the same alloys prepared by CBG, the phases present in the samples produced by ABG were identified by X-ray diffractometry (XRD). This was performed using a Philips 1700 X-ray diffractometer with Cobalt  $K_{\alpha}$  radiation with reference to XRD patterns given in the JCPDS files in the case of well verified equilibrium phases, and to those found in the literature in the case of non-equilibrium and less well defined constituents.

### 3. RESULTS

The representation of solidification microstructure versus growth velocity  $V$  obtained in ABG in Fig.1 is similar to that obtained by CBG [5,6] except that the suppression of primary  $Al_3Fe$  and eutectic transition are displaced to slightly higher growth velocity.

#### 3.1 Binary Al-3.0Fe

The velocity at which the interdendritic  $\alpha Al-Al_3Fe(Eu1)$  to  $\alpha Al-Al_6Fe(Eu2)$  eutectic transition took place was found to be 0.1mm/s under the present solidification conditions, compared to 0.05 to 0.09mm/s in CBG [5]. An essentially flat transition boundary perpendicular to the growth direction was observed across the sample. Detailed observation, Fig.2, revealed that, with an acceleration  $a=3.61 \times 10^{-4} mm/s^2$ , the boundary was quite sharp except for protrusion of a few primary  $\alpha Al$  dendrites into the Eu2 matrix, indicating that there was no intermediate structural change between Eu1 and Eu2. No major differences in the resulting solidification microstructures produced by ABG and CBG were noted except that a few primary  $Al_3Fe$  and  $\alpha Al$  crystals remained even after the Eu1 to Eu2 transition up to 0.29mm/s in ABG.

#### 3.2 Effects of copper and magnesium

Addition of Cu or Mg introduced primary  $\alpha Al$  dendrites over the velocity range tested. In the Cu-containing alloy, primary  $Al_3Fe$  crystals were rarely found and were small in size at  $\leq 0.05 mm/s$ . Eu2 formed as an interdendritic constituent at  $\geq 0.06 mm/s$ . Fig.3 shows typical  $\alpha Al$  dendrites plus interdendritic Eu2 structure from 0.2 to 1.0mm/s over which there was no dramatic structural change except that dendritic and eutectic scales decreased with increasing growth velocity. In contrast, for the Mg-containing alloy, primary  $Al_3Fe$  crystals were found up to 0.6mm/s and Eu2 required  $\geq 0.28 mm/s$  to grow. An interdendritic cellular Eu2 structure similar to that in Cu-containing alloy was observed in the velocity range 0.47 to 1.0mm/s.

#### 3.3 Effect of silicon

$\alpha Al-\alpha AlFeSi$  eutectic (Eu5), was favoured over Eu2 as an interdendritic constituent at  $\geq 0.09 mm/s$  due to the presence of Si. With increasing growth velocity, the morphology of Eu5 changed abruptly twice, at 0.29 and at 0.61mm/s. Fig.4 shows the transition boundary for Eu5 to change morphologically at 0.29mm/s. Figs.5a and b show the typical microstructure of  $\alpha Al$  dendrites plus interdendritic Eu5 at 0.31 and 0.64mm/s respectively, indicating the difference in morphology of the eutectic at the two growth velocities. At high velocity, e.g. 0.62 to 0.84mm/s, Eu5 appeared to grow with a fan-like morphology, Fig 6.

### 3.4 Effect of vanadium

Addition of vanadium introduced a transition from interdendritic Eu1 to  $\alpha\text{Al-Al}_x\text{Fe}$  eutectic (Eu3) at 0.06 to 0.09mm/s. As shown in Fig.7, primary  $\text{Al}_3\text{Fe}$  and  $\alpha\text{Al}$  crystals, individual Eu1, Eu2 and Eu3 grains were all observed over the velocity interval indicating that the Eu1 to Eu3 transition appeared to involve an intermediate phase  $\text{Al}_6\text{Fe}$ . Eu3 exhibited similar morphologies to those in CBG and showed a lamellar to rod-like transition with increasing growth velocity .

## 4. DISCUSSION

Bridgman experiments with either sudden or gradual change in growth velocity (acceleration and deceleration) have been used previously to determine the stable growth morphology and transitions of some eutectics [7-10]. A hysteresis in change in eutectic spacing was observed when changes in growth velocity were imposed [8-10]. The faster the change in growth velocity imposed, the larger the hysteresis. The slightly higher velocities required for suppression of primary  $\text{Al}_3\text{Fe}$  and for the eutectic transition in ABG compared to CBG found in the present study may reflect similar hysteresis effects. It was also found [10] that, under certain solidification conditions in Bridgman growth, some eutectics, such as Ag-Cu, Al-Zn and Al-CuAl<sub>2</sub>, showed little change in their eutectic spacing with continuously increasing growth velocity over a velocity range until to a critical growth velocity at which they reduced their eutectic spacing abruptly. This discontinuity for change in eutectic spacing was not observed for  $\alpha\text{Al-Al}_6\text{Fe}$  and  $\alpha\text{Al-Al}_x\text{Fe}$  eutectics in the present study. The two abrupt morphological transitions found for the  $\alpha\text{Al}-\alpha\text{AlFeSi}$  eutectic, however, demonstrates that this eutectic adjusts its growth behaviour in a discontinuous way to accommodate the change in growth velocity.

In their pioneer investigation on  $\text{Al}_x\text{Fe}$  phase formed in unidirectionally solidified binary hypoeutectic Al-Fe alloys, Young and Clyne [11] have pointed out that  $\text{Al}_x\text{Fe}$  might act as an intermediate phase via which the transition between Eu1 to Eu2 takes place. The present study revealed no other intermetallic during the Eu1 to Eu2 transition under the present solidification conditions. The ABG experiments, however, revealed a Eu1 to Eu3 transition in the V-containing alloy which apparently involves metastable  $\text{Al}_6\text{Fe}$  as an intermediate phase. Todd and Jones [5] observed a similar transition in their study of Al-0.5wt%Fe containing 0.09wt%Si, where the dominant interdendritic intermetallic in Bridgman grown samples changed from  $\text{Al}_3\text{Fe}$  to  $\alpha\text{Al}_x\text{Fe}$  via  $\text{Al}_6\text{Fe}$  with increasing growth velocity.

## 5. CONCLUSIONS

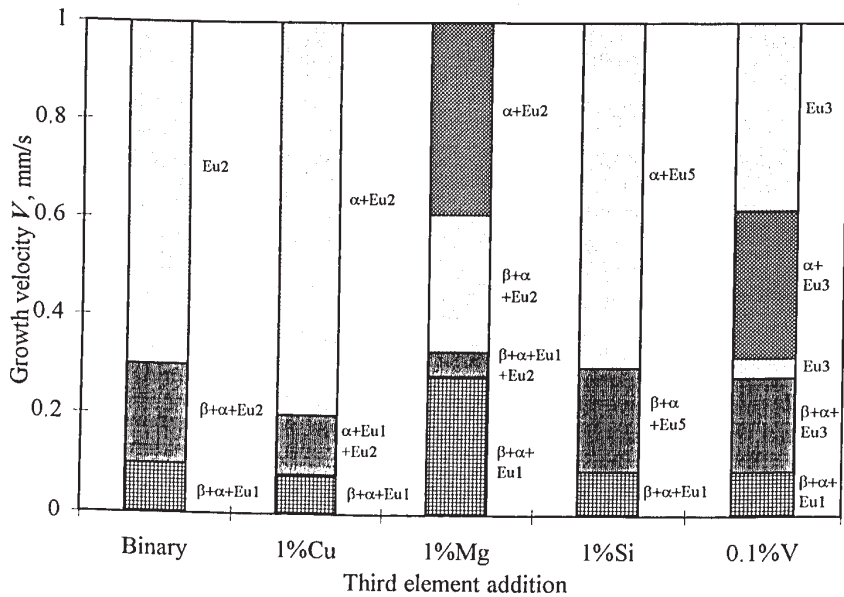
- With an acceleration  $a=3.61\times 10^{-4}\text{mm/s}^2$ , the critical velocity for interdendritic  $\alpha\text{Al-Al}_3\text{Fe}$  to  $\alpha\text{Al-Al}_6\text{Fe}$  eutectic transition in binary Al-3wt%Fe alloy is 0.1mm/s. Addition of 1wt% Mg required  $>0.61$  and  $>0.28\text{mm/s}$  to suppress primary  $\text{Al}_3\text{Fe}$  and to replace  $\alpha\text{Al-Al}_3\text{Fe}$  with  $\alpha\text{Al-Al}_6\text{Fe}$  eutectic respectively.
- Addition of Si or V displaced  $\alpha\text{Al-Al}_3\text{Fe}$  eutectic by  $\alpha\text{Al}-\alpha\text{AlFeSi}$  and  $\alpha\text{Al-Al}_x\text{Fe}$  eutectics respectively at  $\geq 0.09\text{mm/s}$ . The  $\alpha\text{Al-Al}_3\text{Fe}$  to  $\alpha\text{Al-Al}_x\text{Fe}$  eutectic transition introduced by the addition of vanadium occurred in the velocity range 0.06 to 0.09mm/s and appeared to involve  $\text{Al}_6\text{Fe}$  as an intermediate phase.
- $\alpha\text{Al}-\alpha\text{AlFeSi}$  eutectic changes its morphology at 0.29 and 0.62mm/s respectively, indicating that this eutectic alters its growth behaviour discontinuously with a continuous increase in growth velocity.

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Key:  $\beta$ =primary  $Al_3Fe$ ,  $\alpha$ = $\alpha$ Al dendrites, Eu1= $\alpha$ Al- $Al_3Fe$  eutectic, Eu2= $\alpha$ Al- $Al_6Fe$  eutectic, Eu3= $\alpha$ Al- $Al_xFe$  eutectic and Eu5= $\alpha$ Al- $\alpha$ AlFeSi eutectic

Fig.1 Effects of ternary additions on solidification microstructure formation of Al-3wt%Fe in ABG

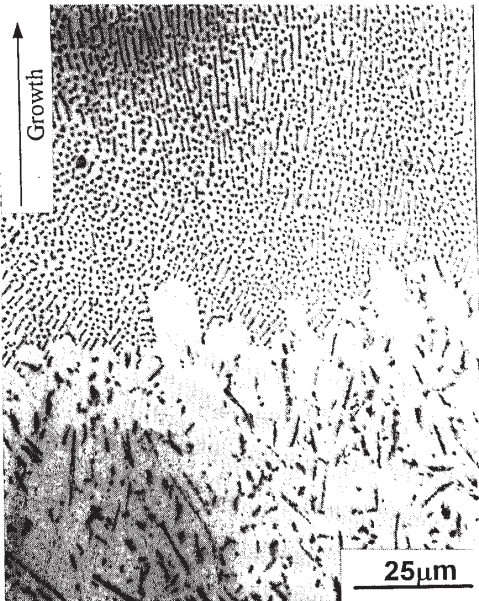


Fig.2 Optical micrograph showing a sharp interdendritic  $\alpha\text{Al}-\text{Al}_3\text{Fe}$  to  $\alpha\text{Al}-\text{Al}_6\text{Fe}$  eutectic transition at 0.1mm/s in the binary alloy (longitudinal section)

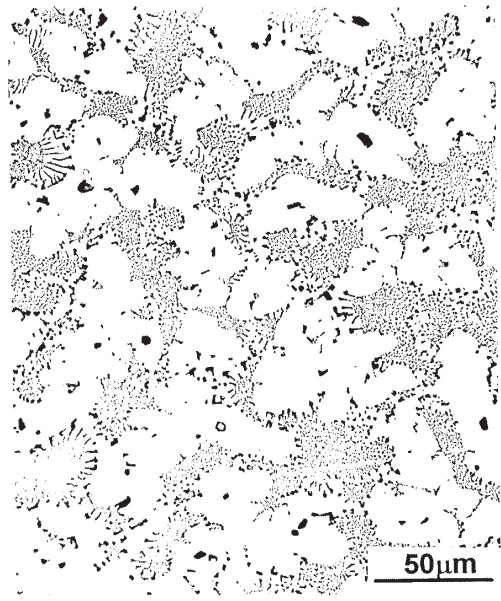


Fig.3 Typical  $\alpha\text{Al}$  dendrites plus  $\alpha\text{Al}-\text{Al}_6\text{Fe}$  eutectic (Eu2) structure formed at 0.55mm/s in Al-2.85Fe-0.96Cu (transverse section)

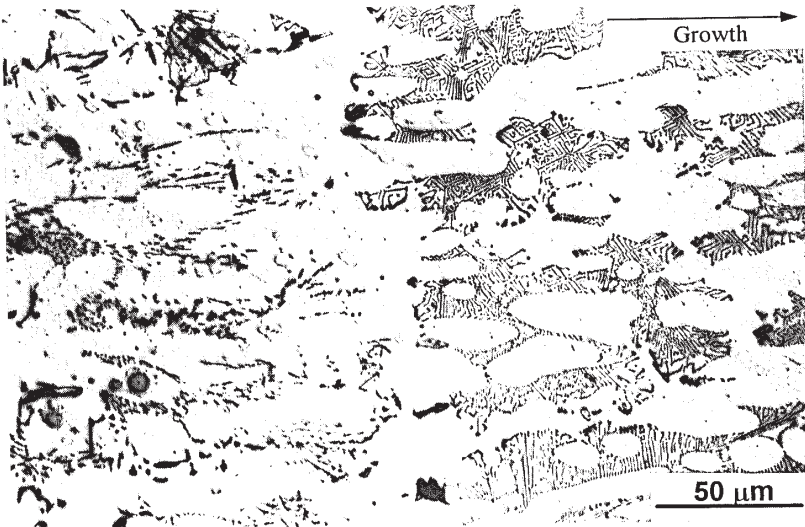


Fig.4 Optical micrograph showing a sharp boundary for a morphological transition of interdendritic  $\alpha\text{Al}-\alpha\text{AlFeSi}$  eutectic at 0.29mm/s in Al-2.91Fe-1.08Si (longitudinal section)

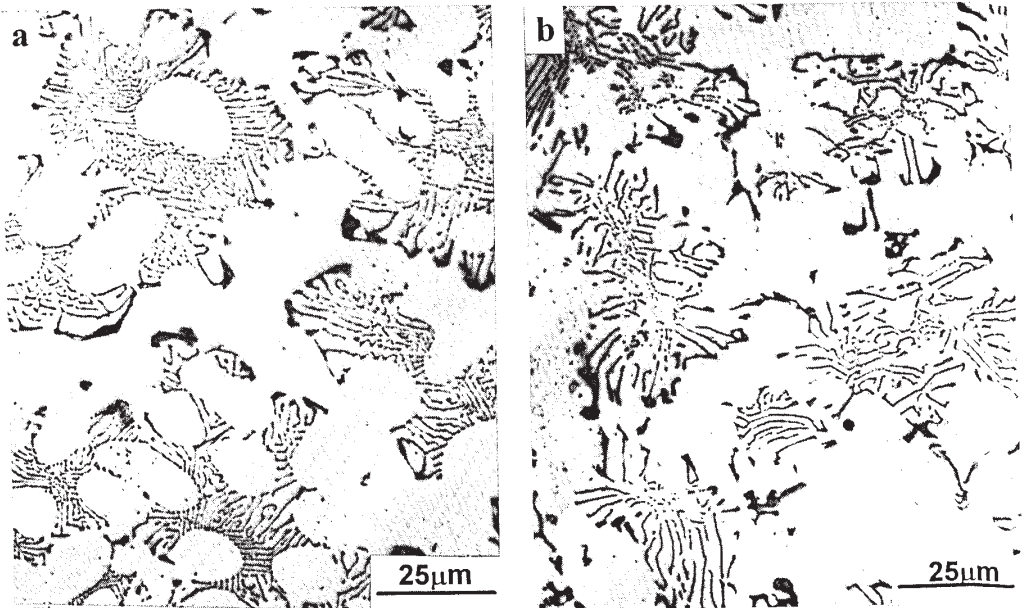


Fig.5 Optical micrographs showing the typical morphologies of interdendritic  $\alpha$ -Al- $\alpha$ -AlFeSi eutectic (Eu5) at (a) 0.31 and (b) 0.64mm/s in Al-2.91Fe-1.08Si (transverse section)

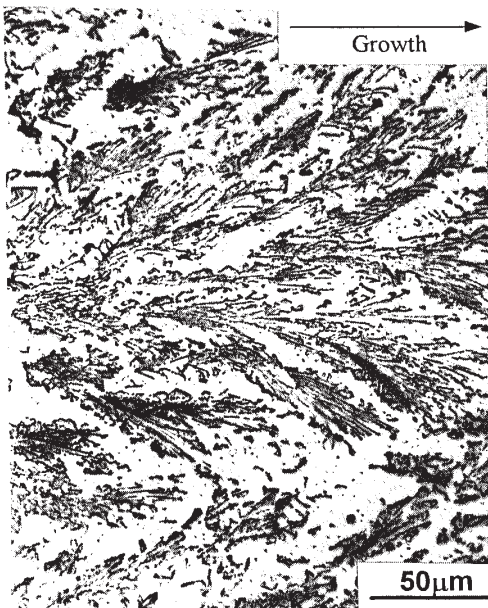


Fig.6 Optical micrograph showing a fan-like morphology for interdendritic  $\alpha$ -Al- $\alpha$ -AlFeSi eutectic (Eu5) at high velocity (0.79mm/s) in Al-2.91Fe-1.08Si (longitudinal section)

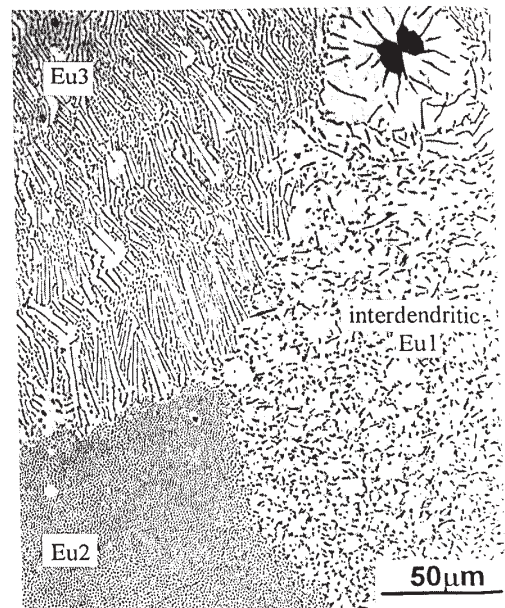


Fig.7 Optical micrograph showing the co-existence of primary  $Al_3Fe$ ,  $\alpha$ -Al dendrite, Eu1, Eu2 and Eu3 at 0.09mm/s in Al-2.85Fe-0.12V (transverse section)