# The Effect of Fe and Mn Content on Coarse Grain Formation During Homogenisation of 6000 Series Alloys

E.D. Sweet, E.S. Charles, M.J. Couper

Comalco Research and Technical Support, Thomastown, Australia

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## Abstract

In 6000 series alloys with very low iron content, grains at least 20 mm long can sometimes form during homogenisation when a few isolated grains begin to grow and become much larger than the surrounding grains. It is likely that the effect of Fe is the result of an absence of fine intermetallic particles, which play a role in the prevention of recrystallisation. It therefore follows that Mn, whose presence also contributes to the formation of intermetallic particles, may affect recrystallisation. For smelters that produce high purity grades, the Fe level in available metal can be particularly low. For this reason it was decided to explore the effects of Fe and Mn on the tendency to coarse grain formation during homogenisation. The results showed that billets with low Fe, low Mn alloy content have increased tendency towards abnormal grain growth during homogenisation.

## 1. Introduction

The presence of coarse grains in either rolling slab or extrusion billet can cause streaking defects and variations in surface brightness in the end product. Often the defects are accentuated by etching, polishing or anodising [1]. Generally the large extrusion ratios created by extrusion can produce a more uniform grain structure than is possible in sheet [1], although multi-hole dies or large profiles can have smaller extrusion ratios, and the presence of corners, junctions and increases in thickness can lead to larger or non-uniform grain structures. Even when the material has recrystallised to produce a fine-grained structure, the existence of particles on the original grain boundaries or the texture of the material can lead to streaking defects. Figure 1 shows the structure of a low Fe containing homogenised 6063 alloy after 3 hours at 570°C, exhibiting non-uniform coarse grain formation.

The present work focuses on the formation of coarse grains in 6000 series extrusion billet during homogenisation, and the effect of Fe and Mn content on its development. Although this phenomenon is not usually observed in conventional 6000 series billet it is claimed that in alloys with very low iron content, such as 6463, there can be enough unpinning during homogenisation processes to cause the growth of coarse grains. This process has variously

been called "discontinuous grain growth", "secondary recrystallisation" [2] or "abnormal grain growth" [3-5]. Abnormal grain growth occurs when a few isolated grains begin to grow and become much larger than neighbouring grains. The larger grains consume the finer neighbouring grains in an attempt to reduce the overall grain surface energy.



Figure 1: Coarse grain growth formed during homogenisation for 3 hours at 570°C (6063 alloy, 0.07 wt% Fe, 0.005 wt% Mn). Complete  $\phi$ 127 mm cross-section shown.

In general, the driving force for abnormal grain growth increases with increasing casting speed, decreasing iron content, increasing homogenisation temperature and holding time, increasing grain refinement and inhomogeneous grain size [3]. Homogenisation affects grain growth since during homogenisation small particles dissolve and large particles coarsen, and the particles become more spherodised. This causes the local driving force for grain growth to exceed the Zener drag from particle pinning [6]. A high temperature increases grain boundary mobility, and grain growth will occur at lower temperatures when the time at temperature is increased. Pre-existing coarser grains, such as those near the billet surface aid nucleation of coarse grains [2,7], since these large grains will grow at the expense of the surrounding smaller grains. Therefore, the coarse grains can be localised in regions around the periphery of the billet. For a more thorough explanation of the various effects on grain growth refer to [3]. Other factors that might cause coarse grain growth during homogenisation include casting stresses [8], mechanical handling of the billets, and hot spots in the furnace such as where the billets contact the support saddles or racks.

Low iron content increases the driving force for coarse grain formation through the absence of fine intermetallic particles, which play a role in the prevention of recrystallisation [2,9,10]. It therefore follows that Mn content, whose presence also contributes to the formation of intermetallic particles and is known to affect recrystallisation, may alter grain growth behaviour. In addition, metallography on billet slices examined previously at Comalco [11] suggested that there was something to be learned from the presence or absence of subgrains in the billet slice. Heavy subgrain formation was observed in the fine grain regions, while the coarse-grain regions were essentially free of subgrains. Thus, it was thought possible that a link exists between the presence of intermetallic particles, the development of subgrains and the formation of coarse grains. These observations are consistent with modelling simulations used to develop a model for abnormal grain growth based on abnormal subgrain growth [12].

It is however presently unknown whether or not the formation of subgrains alters with the Fe and Mn content or whether it is just the visibility of the subgrains that changes. When fine intermetallic particles are present, it encourages homogeneous Mg-Si precipitation. However, in the absence of these Fe/Mn particles, Mg-Si precipitation tends to occur on subgrain boundaries, highlighting the existence of subgrains.

In addition to the effects of Fe and Mn contents, it was decided to determine the effects of homogenisation time on coarse grain formation. Occasional stoppages in the continuous homogenisers can result in longer than standard soak times, and batch homogenisation times are often significantly longer than standard times for continuous homogenisation.

## 2. Experimental Procedures

Four  $\phi$  127 mm 6063 alloy billets were cast at the Comalco Research and Technical Support Centre with nominal composition: 0.48 wt% Mg, 0.40 wt% Si and 0.01 wt% Ti. The four billets were labelled HH, HL, LL and LH with the first alpha representing the Mn level and the second alpha representing the Fe level. The low Mn level was chosen as 0.005 wt% and the high Mn level was chosen as 0.04 wt%, which is a typical upper content for 6000 series alloys. The low Fe level was chosen as 0.07 wt%, and the high Fe level was chosen as 0.18 wt%, which is a typical upper content for standard purity 6000 series alloys (Table 1).

Two 40 mm slices from each alloy were homogenised by heating from room temperature to 570°C in 2.2 hours, soaking for 3 hours at 570°C and controlled cooling at 800°C/h. This cycle was then repeated for a soak time of 15 hours. The orientation of the slices with respect to the furnace racks was noted to test whether the contact influences grain growth behaviour.

The as-cast and homogenised grain structure was examined for each alloy. Each homogenised billet slice was machined on the cut faces to remove the oxide scale formed after homogenising. The slices were then macroetched on both faces with Poultan's reagent for 1 minute to reveal the grain structure. Cross sections were taken from all machined billet slices from both the coarse and fine grain regions when applicable. These samples were hot mounted, ground and polished to 1  $\mu$ m. Final polishing was performed using magnesium oxide powder. These samples were then etched with Kroll's reagent to examine for subgrains.

#### 3. Results

The as-cast microstructures were similar for all four billet compositions, with LH having a slightly larger grain size. Figure 2 shows the as-cast macrostructure. Grain growth (g.g.) was observed in a number of homogenised alloys, and all homogenised billet slices have been ranked from largest grain size to smallest grain size as shown in Table 1. Usually the grain growth was different on either face of the billet slice and the two slices also had variable grain structures. No link was made between the position of grain growth on a slice and the way it was sitting on the furnace racks. Some example photographs of billet slices exhibiting grain growth are included in Figure 3.

No subgrains were present in any of the alloys. All alloys had a light precipitation density except for alloy HH, which had a medium precipitation density.



Figure 2: As-cast macrostructure for alloy 6063 (LL - 6063 alloy, 0.07 wt% Fe, 0.005 wt% Mn). Half  $\phi$ 127 mm cross-section shown.

Table 1: Ranking of Billet Slices							
Alloy And	Soak Time	Grain Growth	Comments				
Slice Number	(Hrs)						
HH – 0.04Mn/0.18Fe; HL– 0.04Mn/0.07Fe; LH – 0.005Mn/0.18Fe; LL – 0.005Mn/0.07Fe							
LL-1	15	Yes	Large g.g. observed on both faces, with small grains scattered throughout the large grains (Figure 3a).				
LL-1	3	Yes	Large g.g. on both faces, which is extensive on face 1 and mainly peripheral for face 2 with only some mid-radius g.g (Figure 3b).				
LL-2	15	Yes	G.g significantly different for both faces: Face 1 showed large peripheral g.g, Face 2 showed small peripheral g.g. and no g.g. in towards the centre (Figure 3c).				
LL-2	3	Yes	Large g.g. on one face only, which is mainly peripheral with some mid-radius g.g (Figure 1).				
HL-1	15	Yes	Medium g.g. with more on face 1 than face 2.				
LH-1 HL-1 HH-1	15 3 15	Yes Yes Yes	Small-medium g.g. on one face 1. Less or no g.g on second face (Figure 3d).				
LH-1	3	V. slight					
LH-2 HL-2 HH-2 HH-1, 2	3, 15 3, 15 15 3	No No No No	Similar grain size for all slices. Note that the grain size for these alloys is similar to the as-cast grain size of all alloys (Figure 2).				



(a) (b) (c) (d) Figure 3: Macrostructures for variants of alloy 6063 homogenised at 570°C for different times (a), (c) LL: 15 hrs, (b) LL: 3 hrs, (d) HL: 3 hrs. Complete  $\phi$ 127 mm cross-section shown. There appears to be a large degree of randomness associated with whether or not coarse grain growth occurred during homogenisation and also with the extent of the coarse grain growth in a particular sample. This randomness was evident by comparing one face of a billet slice to the other or for two slices homogenised under the same condition (for example, compare Figures 1 & 3b or Figures 3a & 3c). Often grain growth was only evident on one face, or the pattern of coarse grains differed from face to face. In many slices, some grain growth was observed on the external billet surface. Cross-sections through the slices confirmed the randomness. The position of coarse grains did not correlate with the orientation of the racks in the homogenisation furnace.

Referring to Table 1, the size of the coarsened grains does not seem to correlate with homogenisation time (3h versus 15h). However, the occurrence of coarse grains does seem to follow a consistent pattern as summarised in Table 2, though many more slices would be required to statistically validate these trends. The likely occurrence of coarse grains increases with reduced Fe only, reduced Mn only and both reduced Fe and Mn. For the two cases of higher Fe, the longer homogenisation time (15h compared to 3h) does seem to have increased the likelihood of coarse grains.

Designation	Mn level	Fe level	3h / 570°C	15h / 570°C
LL	L	Ц	2	2
HL	Н	L	1	1
LH	L	Н	0	1
HH	Н	Н	0	1

Table 2: Occurrence of coarse grains in homogenised slices (number of slices out of two per condition)

Modelling simulations by Holm *et al* [12] showed that the emergence of abnormal subgrains from the initial structure is a random event requiring both a favourable subgrain orientation combined with a favourable neighbourhood for growth. In their work only one or two simulations in ten exhibited abnormal behaviour even when the conditions were set to allow such abnormal growth.

It may be that face-to-face variation and edge-to-edge variation is affected by local changes in alloy content or intermetallic distribution or that edge-to-edge variation is affected by different casting stresses from one billet edge to the other. The work by Holm *et al* [12] would suggest that it is the compositional differences rather than the differences in stress conditions that are having the predominant effect. No stress or surface effects were included in their simulations, whereas boundary mobility was found to be a critical factor. Abnormal growth was found to occur when a subgrain's environment allowed it to grow and its boundary mobility allowed it to grow much more quickly than its neighbours.

Despite the observed randomness, there is a strong indication that the low Mn, low Fe billet slices (LL) have a greater tendency towards grain growth than the other slices. Rios [13] has shown theoretically that the most susceptible microstructure to abnormal grain growth consists of very fine grains pinned by very fine slowly coarsening particles, with a heterogenous particle distribution. A low Mn, low Fe 6063 alloy composition would fit this

condition, given that it will have the lowest volume fraction of intermetallic particles of the four alloys studied. A detailed investigation of the alloy microstructures (size, type and distribution of intermetallics) might provide an explanation for the relative susceptibility of the other alloy versions. The results may indicate that Fe has a stronger influence on preventing susceptibility to grain growth than Mn, but note that the difference in Fe content between the low Fe and high Fe versions (0.11wt%) is greater than the difference in Mn between low Mn and high Mn versions (0.035wt%).

In addition, it was interesting that subgrains were not observed through optical microscopy in any of the samples, regardless of Mn and Fe content, either in fine-grain or coarse-grain regions. Whether or not subgrains would be observed using TEM is not known. This suggests that the presence or otherwise of subgrains visible through optical microscopy might not be as relevant as previously thought.

#### 5. Conclusions

Abnormal grain growth was induced during homogenisation of some 6063 alloy variants at 570°C. However, there was a degree of randomness associated with whether or not coarse grain growth occurred, as well as the size and extent of coarse grain growth. Despite the observed randomness, there is a strong indication that the low Mn, low Fe billet slices (LL) have a greater tendency towards abnormal grain growth than the alloy variants with high Mn and/or high Fe. The results suggest that Fe may have a stronger influence on preventing susceptibility to grain growth than Mn for the levels studied. Under conditions were the likelihood of coarse grains is reduced (high Fe), the results suggest there may be an increased likelihood of coarse grain growth for longer homogenisation times.

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