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THE EFFECTS OF MODIFICATIONS TO COMPOSITION AND PROCESSING ON RECRYSTALLIZATION RESISTANCE OF 6013

B. Thanaboonsombut[1] and T.H. Sanders, Jr.[2]
[1]National Metal and Materials Technology Center, Rajdhevee, Bangkok 10400, Thailand
[2]School of Materials Science and Engineering, Georgia Institute of Technology Atlanta, GA 30332-0245 USA

Abstract

This paper reviews the results of several experimental investigations relating Mn and Fe content, the addition of zirconium, solidification rate, and preheat temperature on the recrystallization resistance of 6013.

Introduction

In another paper published in these preceedings [1] the basic physical metallurgy of 6013 was reviewed. To maintain strength and corrosion resistance, copper, magnesium and silicon levels were fixed. However, by controlling the Fe and Mn, by adding Zr, by controlling the preheat temperature, and by changing solidification parameters, variations in the resistance of this 6XXX alloy can be accomplished.

Experimental Results

To determine the effect of different amounts of iron and manganese on recrystallization resistance of the alloy, several sets of compositional variants were prepared. The amounts of copper, silicon and magnesium were fixed at the nominal levels for 6013 (1.00 wt% Cu, 0.70 wt% Si and 0.87 wt% Mg) for the different variants. As-cast ingots were preheated using a heat treatment similar to what a commercial ingot might experience in a production environment. The ingots were heated at a rate of 50°C/hr to either 500°C or to 560°C and held at that temperature for, 8 hours or 4 hours, respectively.

Preheated ingots were then rolled at 440°C. The ingots were heated rapidly (10°C/minute) to 440°C to minimize precipitation of the major solute elements and rolled to a total of 40, 63, and 80% decrease in thickness after the first, second and third passes, respectively. After each pass the ingot was quenched in water and a section was cut from each ingot. The rest of the ingot was rapidly reheated to 440°C for further rolling. A smaller, longitudinal section of each piece that was cut from the rolled material was then solution heat treated (SHT) in a molten salt bath at 560°C for 10 minutes. The temperatures used for SHT are typically high enough (in this case 560°C) to provide sufficient thermal activation for recrystallization of wrought 6013

560°C) to provide sufficient thermal activation for recrystallization of wrought 6013 products. If any significant driving force is present (i.e. strain energy) recrystallization may occur quickly during the SHT step. A schematic showing the processing steps is given in Figure 1.

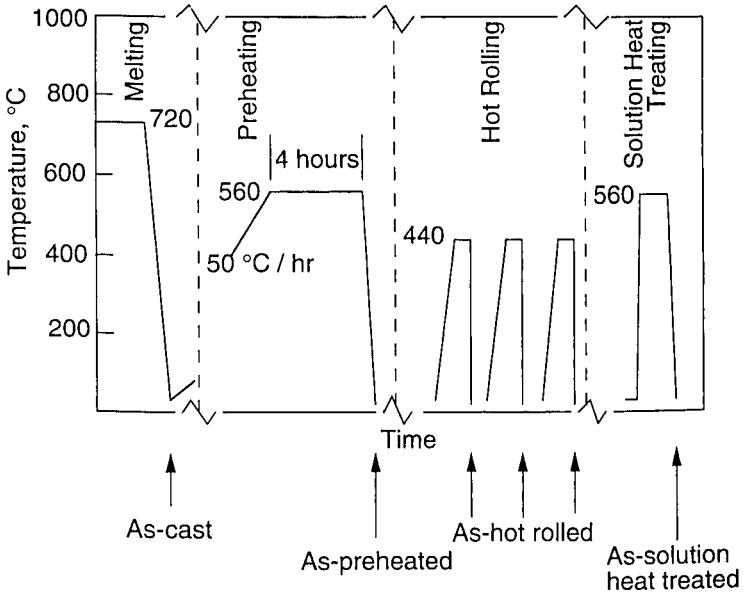

Figure 1 A schematic of the processing steps adopted in this research

Results

Compositional Variants of 6013 (Mn-variant and Fe-free alloys)

As-cast Microstructures. The insoluble phase in the as-cast Fe-bearing alloys is coarse α(AlFeMnSi) phase, whereas the corresponding phase in the as-cast Fe-free alloys is coarse α(AlMnSi) phase [2]. Although the coarse α(AlMnSi) particles in the Fe-free alloy have a slightly smaller particle size and a substantially lower volume fraction than the α(AlFeMnSi) particles in the Fe-bearing alloy, their sizes are sufficiently large (much greater than 1 micron, in the longest dimension) to act as a preferential site for nucleation of recrystallized grains [3,4]. In both cases, the volume fraction of these particles increases substantially with increasing manganese content. However, for alloys containing comparable levels of manganese, the volume fraction of the coarse α(AlMnSi) particles in an Fe-free alloy is substantially less than that of the α(AlFeMnSi) particles in Fe-bearing alloys, Figure 2.

The composition of the coarse α(AlFeMnSi) phase in the Mn- and Fe-variants is a function of the iron and the manganese content, Figure 3. The composition of this

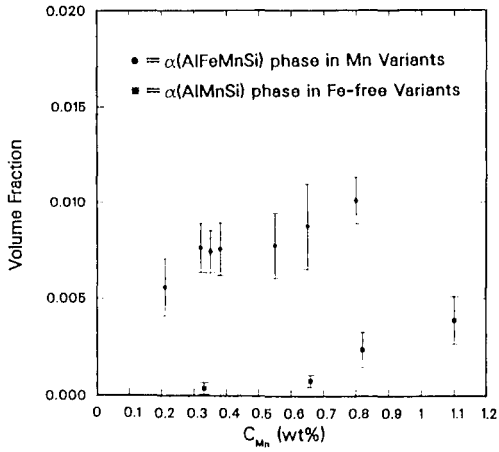

Figure 2 A plot of volume fraction of coarse constituent α(AlFeMnSi) phase in Mn-variant 6013 and α(AlMnSi) phase in Fe-free 6013 as a function of manganese content.

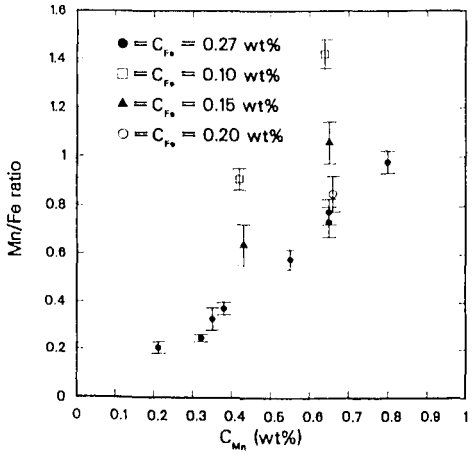

Figure 3 A plot showing the Mn/Fe Ratio of the α (AlFeMnSi) for Mn-and Fe-variants of 6013.

phase can be characterized by the Mn/Fe ratio, which is the relative amount (in atomic percent) of manganese and iron present in the particles. The tendency for manganese to form this phase increases as the relative amount of manganese in the alloy is increased.

The Mn/Fe ratio in the coarse α(AlFeMnSi) phase is a useful parameter since it permits one to estimate the volume fraction of this phase and the amount of manganese retained in supersaturated solution after solidification [2,5]. These two quantities are significant since the coarse phase can accelerate recrystallization and the fine phase can retard recrystallization. If the composition and the volume fraction of the α(AlFeMnSi) phase are known, the amount of manganese that forms this phase and the amount that remains in supersaturated solid solution can be estimated. The only assumption required is that manganese participates in the formation of only the coarse α(AlFeMnSi) phase and the rest remains in supersaturated solid solution. As the manganese content of the alloy increases, both the amount of manganese in the

coarse α(AlFeMnSi) phase and that in the solid solution increases, Figure 4. An increase in the amount of manganese in solid solution would lead to an increase in the volume fraction f, and hence the f/r ratio, of the Mn-bearing dispersoids in the preheated ingots. This increase in f should lead to an increase in recrystallization resistance. However, the overall resistance may be reduced since the volume fraction of recrystallization stimulating particles also increases.

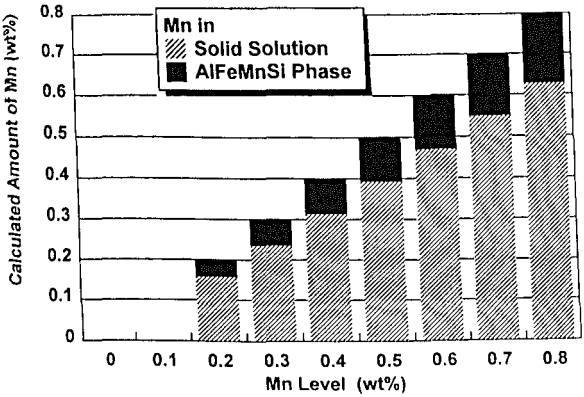

Figure 4 Estimate of the amount of manganese in the coarse α (AlFeMnSi) phase and that retained in solid solution of Mn-variant 6013.

Preheated Microstructures. A standard preheat, like what an ingot might experience in a commercial operation, can be approximated in the laboratory using a ramp heating rate of 50 °C/hr to 560 °C followed by an isothermal hold at 560 °C for 4 hours. The preheated microstructure consists of the coarse α(AlFeMnSi) phase and the Mn-bearing α(AlMnSi) dispersoids. During preheating, the coarse α(AlFeMnSi) constituent particles tend to spheroidize, but their volume fraction and composition remain essentially unchanged [2,5].

Hot Rolled and Solution Heat Treated Microstructures. As the amount of manganese is increased from 0.2 to 0.8 wt%, and if iron is held at 0.27 wt%, the degree of recrystallization decreases with increasing manganese content for a given level of deformation [2]. Figure 5 shows that as the amount of deformation is increased from 40 to 63%, more manganese is required to suppress recrystallization for manganese contents less than 0.65 wt%. At 80% deformation, manganese contents as high as 0.8 wt% are insufficient to suppress recrystallization.

Recrystallized grains are observed to form preferentially at the coarse particles more frequently at high strains although they also occur at low strains. Thus, the increase in recrystallized volume fraction is likely due to the increased deformation in the matrix and to the formation of deformation zones around the coarse α(AlFeMnSi) particles.

It has been shown that the volume fraction of coarse particles has an effect on the recrystallization behavior of aluminum alloy products [6,7]. Since there is a substantial volume fraction of coarse iron-bearing phases in 6013, removing iron from

6013 should increase the recrystallization resistance of the alloy. Figure 6 is a plot of the dependence of volume fraction of recrystallized grains as a function of manganese content in the Fe-free variants of 6013. A comparison of Figure 6 with Figure 5 shows that, without iron, less manganese is required to suppress recrystallization. The decrease in the degree of recrystallization found in the Fe-free variants is due to the small volume fraction of coarse phases and higher amount of Mn-dispersoids found in the Fe-free alloys for a given manganese level. It should be noted that, however, the size of recrystallized grains in Fe-free alloys is significantly larger than that in a corresponding Fe-bearing alloys [8].

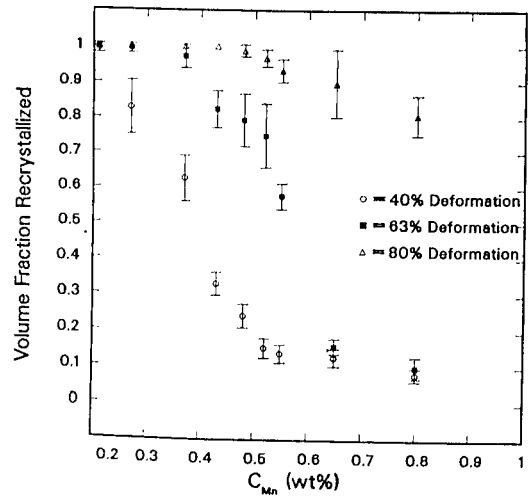

Figure 5 A plot of the Volume fraction % recrystallized grains as a function of manganese content in the Mn-variant 6013 containing 0.27 wt%Fe.

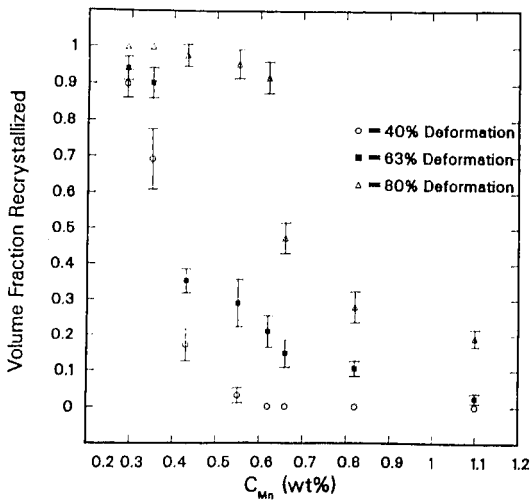

Figure 6 Volume fraction % recrystallized grains as a function of manganese content in the Fe-free 6013.

Effects of Zirconium Additions to 6013

Zirconium is often added to 7XXX series aluminum alloys to suppress recrystallization. In 7XXX series alloys, zirconium forms metastable $L1_2$-Al_3Zr dispersoids. These particles are coherent and very fine (less than 0.03 microns in diameter). However, the presence of silicon tends to destabilize the $L1_2$ phase and the DO_{22} ($Al_{14}SiZr_5$) forms [5,9]. The DO_{22} phase is non-coherent and much coarser (0.1-0.3 microns); thus it is not as effective as the $L1_2$ phase in retarding recrystallization. Elements like lithium tend to stabilize the $L1_2$ phase field and hence many of the new generation aluminum-lithium alloys contain zirconium when an unrecrystallized microstructure is desired.

Mn-bearing 6013 + Zr The Mn-bearing 6013 + Zr samples are examined in order to characterize the effect of zirconium content on the recrystallization resistance of the alloys. The amount of zirconium is varied from 0.00 to 0.10 wt%. The volume fraction recrystallized of the alloys subjected to the standard preheat treatment (560 °C 4 hours), hot rolled and solution heat treated is shown in Figure 7. For a given amount of deformation, the general trend is an increase in recrystallization resistance as the zirconium content increases. This effect is most likely due to an increase in volume fraction of Zr-bearing dispersoids as the amount of zirconium increases. However, samples rolled to 80% deformation and annealed showed negligible resistance to recrystallization, regardless of the zirconium level.

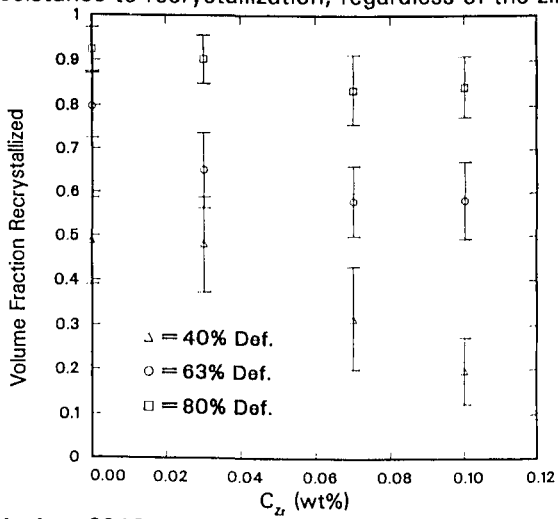

Figure 7 Volume fraction of recrystallized grains as a function of zirconium content in Mn-bearing 6013+Zr alloys subjected to standard preheat.

Mn-free 6013 + 0.10 Zr. Since the presence of zirconium in Mn-bearing 6013 can contribute significantly to the recrystallization resistance of the alloy, it is important to know how Zr-bearing dispersoids alone affect recrystallization. In order to determine the effects of Zr-bearing dispersoids in 6013, the recrystallization behavior of Mn-free 6013 + 0.10Zr was studied. The alloy variant was processed in the same manner as that of Mn-bearing 6013+Zr. The recrystallization behavior of the Mn-free 6013+0.10Zr subjected to standard preheat treatment is shown in Figure 8. The data from the Mn-bearing 6013+0.10Zr are also included for comparison. It is seen that

Mn-free 6013+0.10Zr shows negligible recrystallization resistance. Even at relatively low strain (40% reduction in thickness) the microstructure is almost fully recrystallized (more than 80% by volume fraction). This result suggests that the volume fraction of the Zr-bearing dispersoids in this sample is too small to significantly suppress recrystallization.

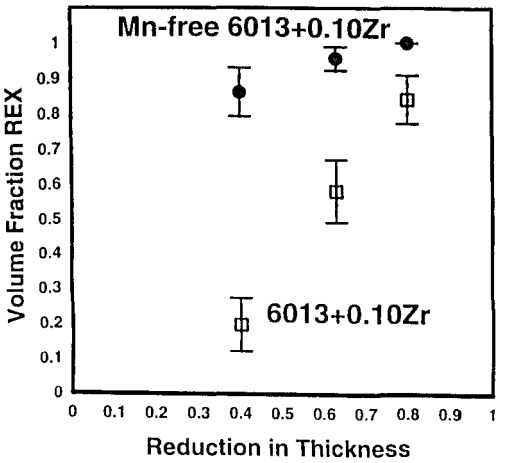

Figure 8 Volume fraction of recrystallized grains as a function of reduction in thickness for Mn-bearing 6013+0.10Zr and Mn-free 6013+Zr alloys subjected to standard preheat.

Si-variant alloys. At this point, an important question regarding the interaction between zirconium and silicon in this alloy system arises: At what level of silicon is the formation of the metastable $L1_2$-Al_3Zr phase suppressed? To answer this question, alloys containing 0.90 wt%Mg, 1.00 wt%Cu and 0.12 wt%Zr with varying amount of silicon were prepared. The amount of silicon used was 0.00, 0.06, 0.12, 0.18, 0.25, 0.40, 0.50 and 0.70 wt%. The as-cast ingots were preheated at 500 °C for 8 hours (heating rate of 50 °C/sec), hot rolled at 440°C to 40% reduction in thickness, and solution heat treated at 560°C for 5 minutes.

The volume fraction recrystallized of this set of alloys as a function of silicon content is shown in Figure 9. The recrystallization resistance varies dramatically with silicon content. Solution heat treated microstructures are fully recrystallized for silicon-free alloy, unrecrystallized (volume fraction of recrystallized grains is less than 40%) for silicon level approximately 0.18wt% and partially recrystallized for other silicon levels. The role of silicon in this alloy (Al-0.9Mn-1.0Cu-0.12Zr) is as follows. As silicon is added (in the range of 0 to 0.18%), the $L1_2$-Al_3Zr phase begins to form. Increasing the silicon content increases the volume fraction of the $L1_2$ phase. With the increase in the volume fraction of the $L1_2$-Al_3Zr phase, there is a corresponding increase in the recrystallization resistance. However, at some critical level, the $Al_{14}SiZr_6$ (DO_{22}) phase begins to form and with further increases in the silicon content there is an increase in volume fraction of the DO_{22} phase and a decrease in volume fraction of the $L1_2$ phase. The result is a decrease in the recrystallization resistance with increasing silicon content (from 0.18% to 0.70%).

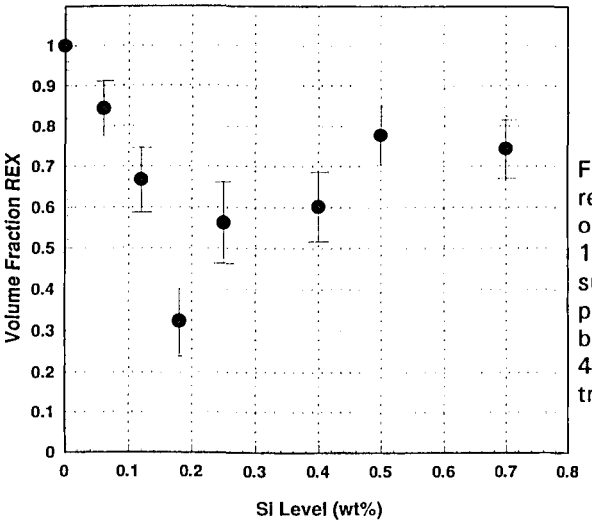

Figure 9 Volume fraction of recrystallized grains as a function of silicon content in Al-0.9Mg-1.0Cu-0.12Zr (Si-variant) alloys subjected to low temperature preheat (560 °C for 8 hr), followed by 40% reduction in thickness at 440 °C, and then solution heat treated at 560 °C for 5 min.

Effect of Preheat Temperature

A low temperature preheat (500°C 8hr) was also used to determine the effect of preheat temperature on the microstructural features and the recrystallization behavior of the alloys.

Preheated Microstructure.

The microstructure of an ingot subjected to low temperature preheat differs from that subjected to standard preheat as follows. The coarse Mg_2Si and α(AlFeMnSi) particles are essentially unaffected by low temperature preheat. Thus, ingot subjected to low temperature preheat consists of coarse Mg_2Si particles in addition to the Fe-bearing phase. More importantly, the Mn-bearing dispersoids in samples subjected to low temperature preheat (approximately 0.08 microns) are significantly finer than those in samples subjected to standard preheat (approximately 0.15 microns). A similar result is also observed for the Zr-bearing dispersoids in Mn-free + 0.10Zr samples. In both cases, the larger size of the Mn- and/or Zr-bearing dispersoids in the samples subjected to standard preheat is attributed to a faster coarsening rate at higher preheat temperature.

Hot Rolled and Solution Heat Treated Microstructure.

The volume fraction recrystallized of the Mn-bearing 6013+Zr alloys subjected to low preheat treatment (500 °C 8 hours) is shown in Figure 10. The general trend is an increase in recrystallization resistance as the zirconium content increased. The major difference of the effect of preheat temperature on the recrystallization behavior is that, for a given amount of deformation and zirconium content, the samples subjected to prior low temperature preheat exhibit greater degree of recrystallization

resistance compared to those subjected to standard preheat. This result is expected since, for a given composition and amount of deformation, the dispersoids in the former case are finer. A smaller size of dispersoids will lead to a higher f/r ratio (or equivalently, higher number density) for a given volume fraction and, hence, results in an increase in the recrystallization resistance of the alloys.

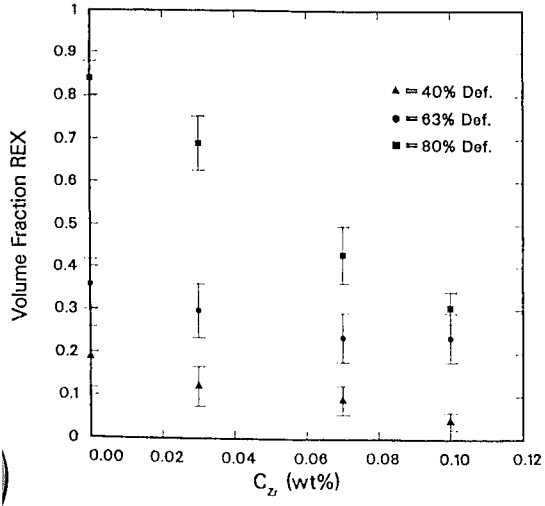

Figure 10 Volume fraction of recrystallized as a function of zirconium contents in Mn-bearing 6013 + Zr alloys subjected to low temperature preheat (560 °C for 8 hr).

Effect of Cooling Rate from the Melt

Alloy 6013 with nominal composition was also cast at different cooling rate to study the effect of cooling rate on microstructural features and recrystallization behavior.

As-cast Microstructure

The scale of the microstructure (as characterized by dendrite arm spacing, segregate spacing and size of constituent α(AlFeMnSi) particles) decrease significantly with increasing cooling rate from the melt. The similarity in the trend for these parameters is attributed to the fact that particle size in the castings as well as the dendrite arm spacing and the segregate spacing, is controlled primarily by growth conditions during solidification of the melt [10].

Figure 11 shows a normalized frequency distribution of the maximum dimension of the α(AlFeMnSi) constituents as a function of cooling rate [5]. In this work the cooling rate from the melt is altered by changing the temperature of the bookmold used in ingot casting. The mold temperature was varied from 25 to 600 °C. A higher mold temperature will lead to a lower cooling rate from the melt, and vice versa. For example, the maximum dimension of the coarse constituent particles in the sample cast at 3.0 °C/sec (corresponding to a mold temperature of 25 °C) ranges from 20 to 80 microns, with the average dimension being approximately 40 microns. The maximum dimension of these particles in sample cast at 0.2 °C/sec (corresponding to

a mold at 600° C), however, spans over a broader range from 80 to 240 microns, with the average dimension being approximately 180 microns.

The Mn/Fe ratio of the coarse α(AlFeMnSi) phase is also a function of cooling rate, Figure 12. This parameter decreases as the cooling rate increased, indicating that there is less manganese in the coarse α(AlFeMnSi) phase and, hence, there should be more manganese in the solid solution in samples cast at higher rate. An increase in the amount of manganese in solution with increasing cooling rate is also observed in alloy 3004 cast at cooling rate within the range of 0.1 to 28 °C/sec [10]. The amount of manganese in solution and that in the coarse α(AlFeMnSi) phase is plotted in Figure 13. It is seen that the amount of manganese in solution slightly increases with increasing cooling rate, and hence the volume fraction of the Mn-bearing dispersoids is expected to be greater in the samples cast at higher cooling rate.

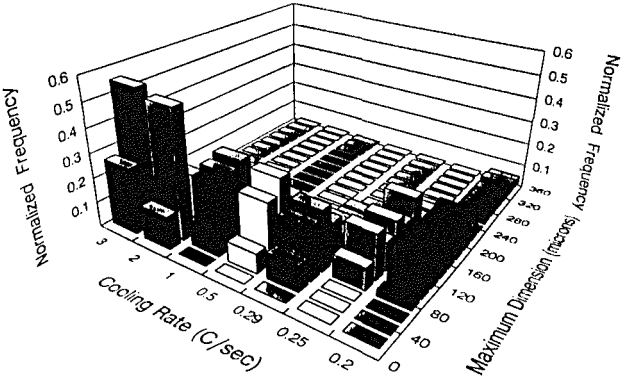

Figure 11 Plot of the normalized frequency distribution of the maximum dimension of coarse α (AlFeMnSi) phase as a function of cooling rate.

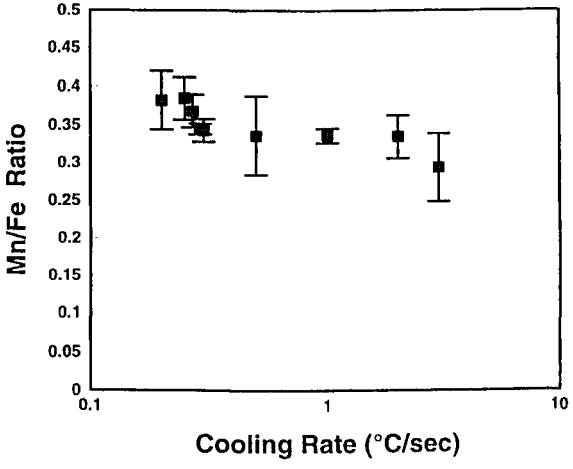

Figure 12 Mn/Fe ratio of the α(AlFeMnSi) phase in samples of 6013 cast at different cooling rates.

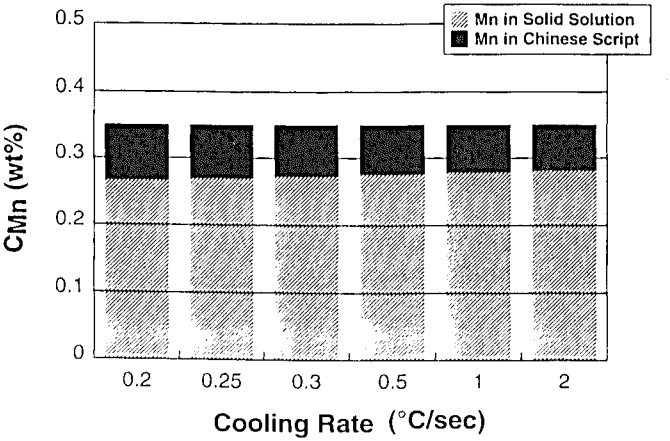

Figure 13 Calculated amount of manganese in supersaturated solid solution and in the coarse α (AlFeMnSi) phase in samples of 6013 cast at different cooling rates.

Hot Rolled and Solution Heat Treated Microstructures.

The recrystallization behavior of the solution heat treated samples are shown as a function of cooling rate in Figure 14. Samples rolled at 80% reduction in thickness show negligible resistance to recrystallization, regardless of the cooling rate. It should be noted that, in this set of samples, although fully recrystallized microstructures are obtained in the samples cast at high cooling rate, a few small elongated unrecrystallized grains can still be observed in the sample cast at the lowest cooling rate (0.2 °C/sec).

The recrystallization behavior of solution heat treated samples previously rolled to 40% and 63% reduction in thickness show unexpected results. The recrystallized

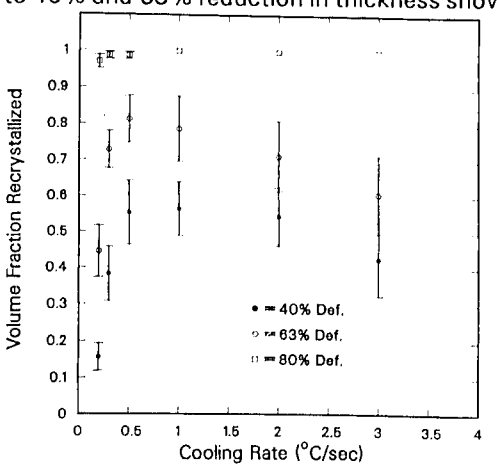

Figure 14 Volume fraction of recrystallized grains in samples cast at different cooling rates.

volume fraction vs. cooling rate curves exhibit maxima in an intermediate range of cooling rate. The volume fraction recrystallized for samples rolled at 40% deformation increases from 15% (0.2 °C/sec) to 53% (1.0 °C/sec) and then decreases to 42% (3 °C/sec), Figure 14. Similar trend is also observed for samples rolled to 63% reduction in thickness.

Two most obvious factors that influence the recrystallization behavior of these samples are (a) the initial grain size and (b) the amount of manganese in supersaturated solid solution. Ingots cast at higher cooling rate have finer microstructure and, hence, greater grain boundary areas per unit volume. Since grain boundary is potential site for nucleation of recrystallized grain, an increase in grain boundary area per unit volume will tend to increase the volume fraction of recrystallized grains. On the other hand, it is found that the amount of manganese in supersaturated solid solution slightly increases with cooling rate; thus ingot cast at higher cooling rate is expected to have higher volume fraction of the Mn-bearing dispersoids. Although this argument may agree qualitatively with the trend of the observed results, a closer inspection of Figure 13 shows that the increase in the amount of manganese in solid solution within the cooling rate range investigated is very small. In fact, experimental results from previous work [2,5,8] suggests that this increase may not be the sole reason for a significant decrease in volume fraction recrystallized at intermediate cooling rates for samples deformed at 40% and 63% reduction in thickness. It is possible, therefore, that there exists another factor responsible for the recrystallization behavior in addition to the effects of the initial grain size and the amount of manganese in solid solution.

There is another important factor that strongly influences the recrystallization behavior of 6013 and has not yet been considered. This factor is the size and the number density of the coarse constituent particles. As experimental results indicated, the size and the number of the coarse constituent α(AlFeMnSi) particles change significantly with the cooling rate from the melt. Thus, this factor may be responsible for the observed recrystallization behavior of the alloy. In order to appreciate the significance of this factor, it is necessary to discuss the role of the particle size during recrystallization in some detail. Generally, coarse particles of size larger than 1 micron are said to be effective in accelerating recrystallization by providing preferential nucleation sites in the region adjacent to them . This occurs because the matrix around these particles tend to be highly strained during deformation, the so-called deformation zone [6,7]. A more precise description of this effect, however, requires that the size of these coarse particles must exceed a certain critical value in order for a sufficiently strained region to be created. This critical size is a function of the deformation conditions, including strain and strain rate and temperature at which the deformation is performed [3,4,6,7]. Experimental observation and theoretical consideration [5] suggests that most of the coarse constituent α(AlFeMnSi) particles in samples cast at relatively high cooling rates (2-3 °C/sec) are not sufficiently large to create deformation zones under the deformation conditions used in this investigation. In other words, these particles are ineffective in accelerating recrystallization. The particles in samples cast at lower rate (0.2-1.0 °C/sec), on the other hand, were sufficiently large and, hence, particle stimulated nucleation occured in these samples. Thus, the role of each of the microstructural features affected by cooling rate along with the deformation conditions are combined in a complicated

manner to influence the recrystallization behavior of the alloy.

Conclusions

1. For a given iron level, increasing the manganese content of the alloy increases the volume fraction of both the coarse constituent α(AlFeMnSi) particles and the fine Mn-bearing dispersoids. However, the effect of increasing the volume fraction of the dispersoids offsets the effect of increasing the number of coarse particles; thus, the recrystallization resistance of the alloy is increased for a given amount of deformation.

2. Reducing the iron content reduces the volume fraction of the coarse constituent α(AlFeMnSi) particles. With fewer coarse particles, the extent of particle stimulated nucleation is diminished and hence the recrystallization resistance of the alloy is increased for a given amount of deformation.

3. The addition of zirconium to 6013 leads to the formation of $Al_{14}SiZr_5$ dispersoids during preheat treatment. Increasing the zirconium content results in an increasing volume fraction of this phase. These particles, however, are of comparable in size to the Mn-bearing dispersoids, but their volume fraction is much lower. Hence, adding zirconium to 6XXX alloy originally containing manganese merely provides higher total volume fraction of dispersoids to retard recrystallization.

4. Increasing cooling rate from the melt results in the following changes in the as-cast microstructural features: (a) decreased ingot grain size, (b) increased supersaturation of manganese, and (c) increased number density of the constituent α(AlFeMnSi) but decreased size. These factors combine to affect the alloy final microstructure, in general, and the recrystallization resistance, in particular.

5. Low temperature preheat (500°C 8 hours) leads to a greater recrystallization resistance as compared to standard preheat (560°C 4 hours) due to a higher density of finer dispersoids.

Acknowledgements

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References

1. B. Thanaboonsombut and T.H. Sanders, Jr. "A Review of the Physical Metallurgy of 6013", these proceedings.

2. R.A. Jeniski , Jr., B. Thanaboonsombut, and T.H. Sanders, Jr., "The Effect of Iron and Manganese on the Recrystallization Behavior of Hot Rolled and Solution Heat Treated Aluminum Alloy 6013," To be published, Met. Trans.

3. F.J.Humphreys and P.N.Kalu, Risø Proceedings, p.385, 1986.

4. F.J.Humphreys and P.N.Kalu, "Dislocation-Particle Interaction during High Temperature Deformation of Two-phase Aluminum Alloys," Acta Metall., Vol.35, No.12, p.2815-2829, 1987.

5. Thanaboonsombut B., PhD Thesis, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA, 1993.

6. Humphreys, F.J. "Recrystallization mechanisms in two-phase alloys," Metal Science, (March-April 1979):136-145.

7. F.J.Humphreys, "Nucleation of Recrystallization in Metals and Alloys with Large Particles", in Recrystallization and Grain Growth in Multiphase and Particle-containing Materials, Proceedings of the 1st Risø International Symposium on Metallurgy and Materials Science, Sept 9-12, 1980.

8. Jeniski Jr, Richard A., Master Thesis, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA, 1993.

9. B.Thanaboonsombut, G. Lapasset and T.H.Sanders,Jr., "The Effect of Zirconium Addition on the Recrystallization Behavior of Hot Rolled and Solution Heat Treated Aluminum Alloy 6013," To be published.

10. P.N.Anyalebechi, T.N.Rouns and R.E.Sanders,Jr., "Effects of Cooling Rate and Grain Refining on Constituent Phase Particle Size in As-cast 3004 Alloy," Light Metals 1991 Edited by Elwin L. Rooy, The Minerals, Metals & Materials Society, 1990.