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Phase Selection and Phase Transformation in Eutectic Iron-bearing Particles in a DC-Cast AA5182 Alloy

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Abstract

The as-cast intermetallic primary particles and their evolution during preheat treatment in a commercial DC-cast AA5182 alloy have been studied. The dominant iron-bearing primary particles in the as-cast alloy have been identified to be Al_m(Fe,Mn) and Al_3(Fe, Mn) instead of Al_3(Fe, Mn) and Al_6(Fe,Mn).The amount of each type of iron bearing particles varies with local cooling rate over the cross section of the ingot. A eutectoid phase transformation from Al_m(Fe,Mn) to Al_3(Fe, Mn) and Al during preheat treatment has been observed by TEM and FEG-SEM. The influence of heating temperature and homogenization time on the phase transformation has also been studied.

1. Introduction

AA5182 alloy is an important commercial aluminium alloy with high strength, high ductility, high corrosion resistance and good formability. It has been widely used for the manufacture of can ends and automobile components. In recent years, it has also been considered for car body sheet [1-3]. The types, size, morphology and distribution of intermetallic primary particles, which have a strong influence on the recrystallization, texture and formability of the alloy, are important features of the as-cast structure of the alloy. The dominant primary particles formed during solidification of commercial AA5182 alloy have been considered to be Al_3(Fe, Mn) (or $Al_{13}(Fe,Mn)_4$), Al_6(Fe, Mn) and Mg_2Si phases[2-8]. A small fraction of Al_8Mg_5 particles can also be found in the alloy [8].

It has been found by Flood et al. that the types of the primary iron-bearing particles and amount of each phase formed during solidification are dependent upon the composition (Fe/Si ratio) in the alloy [5]. Low Fe/Si ratio favors the formation of α-Al(Mn,Fe)Si phase. They also found that there is a phase transformation from Al_3(Fe,Mn) to Al_6(Fe,Mn) during preheat treatment. However, in contrast to the results from the above references, the dominant iron bearing particles in a commercial AA5182 alloy have been identified to be Al_m(Fe,Mn) and Al_3(Fe, Mn) in the present work. The principle for the phase selection of the intermetallic primary particles and phase transformation of the Al_m(Fe,Mn) phase is dicussed.

2. Experimental

The material used in this work is a commercial DC-cast AA5182 rolling ingot with dimensions 510mm thick and 1880mm in width. Chemical composition of the alloy is, in wt%: Mg 4.12, Mn 0.22, Fe 0.36, Si 0.19, Cu 0.0006, Ti 0.02 and Al bal. After mechanical polishing, the intermetallic particles on different locations of the ingot were observed by optical microscopy and scanning electron microscopy (SEM). Preheat treatment was performed in an air circulation furnace with a temperature accuracy of ±2°C. The as-cast samples were heated to 470 and 520 °C with a heating rate of 50 °C/h, respectively, and then homogenized for different times. Isothermal heat treatments were also conducted for the cast samples at 470 and 520 °C. Samples were quenched into water at different temperatures during heating and after different homogenization times. In order to keep the heat-treated samples to have the same initial as-cast structure, all the samples used for heat treatment were taken from locations with 70mm distance to the skin of the ingot along the half width line. A JXA-8900R micro probe was used to measure the composition of the intermetallic particles. A Hitach-430 field emission gun scanning electron microscope (FEG-SEM) was used to observe the morphology of the primary particles after heat treatment. The size characteristics and area fraction of the intermetallic particles were measured using an image analysis program KS300.

TEM foils were prepared by ion milling of 3mm disks cut from the ground and polished 0.2mm thick foils. TEM foils were observed in a JEOL 2010 TEM at 200 kv.

3. Results and Discussion

3.1 Solidification Structure

The solidification structure of the ingot is shown in Figure 1 (a) and (b). The cast ingot has an equiaxed dendrite structure. There are mainly two kinds of primary particles located on the grain boundaries: the white block or plate shaped iron-bearing particles and the dark skeletal Mg_2Si phase. The area fractions of the iron-bearing particles and Mg_2Si particles have been measured to be about 0.8±0.1% and 0.4±0.05%, respectively.

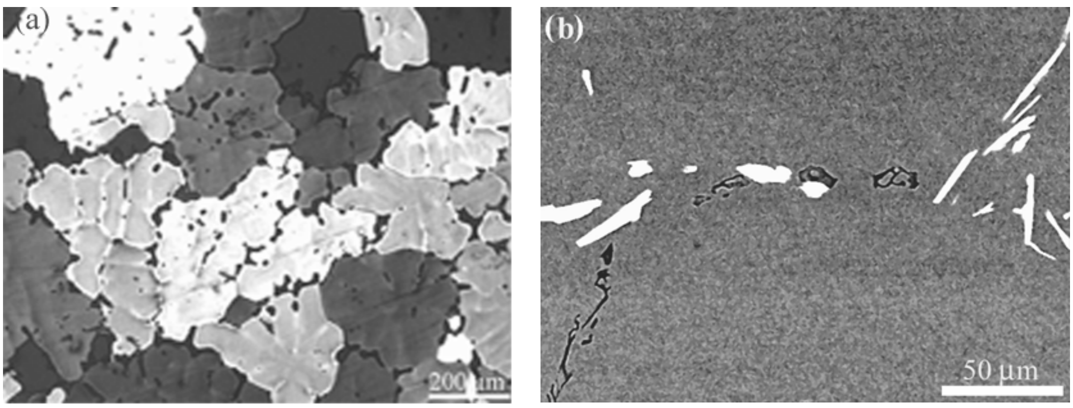

Figure: 1 Dendrite structure (a) and morphology of the primary particles (b) in the as-cast 5182 ingot.

3.2 Identification of Iron-bearing Particles:

The types of the iron bearing particles have been identified by composition of the particles measured by electron microprobe. In order to minimize the contribution of the Al matrix to the composition of the particles, only large particles, whose breadth is larger than 2 μm, are measured. For each sample, 20 particles are selected randomly and measured. The iron- bearing primary particles in commercial 5182 alloys have been previously considered to be Al_6(Fe,Mn) and Al_3(Fe,Mn) [2-8]. However, no Al_6(Fe,Mn) is found in the present alloy. Except for the Al_3(Fe,Mn) particles, there are a large fraction of iron-bearing particles, in whose compositions the atomic ratios between Al and (Fe+Mn) are in the range of 4.1-4.7. This composition is very close to the constitution of the Al_mFe phase with m=4.0-4.4, which has been found recently in Al-Fe-Si alloys [10-12]. This suggests that Al_m(Fe,Mn) is one of the dominant iron-bearing primary particle in the present DC-cast 5182 alloy. The typical compositions of the two types of particles are shown in Table 1.

Table: 1: Chemical composition of the iron-bearing primary particles in the as-cast ingot, wt.%.

Type	Al	Mg	Mn	Fe	Si	Atomic ratio Al/(Mn+Fe)
Al_m(Fe,Mn)	67.047	0.414	3.114	30.021	0.433	4.19
Al_3(Fe,Mn)	62.429	0.093	4.598	32.918	0.506	3.44

The types of the primary particles have also been confirmed by selected area electron diffraction pattern (SADP) and EDX analysis on TEM. Figure 2 shows a SADP of Al_m(Fe,Mn) particle, indicating that the particle has a body centered tetragonal unit cell with a=0.884 nm, c=2.160 nm [13]. In the diffraction patterns, many extra reflection spots parallel to the [110] direction can be observed. These extra reflection spots are due to the faults of stacking sequence in the crystal [12,13].

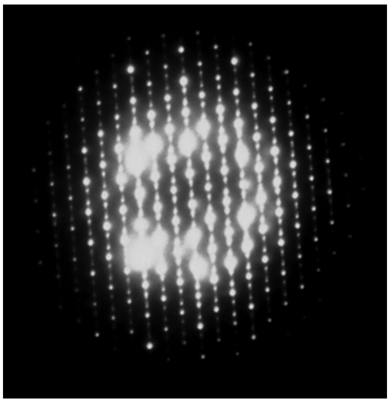

Figure 2: Selected area diffraction pattern (SADP) of the Al_m(Fe,Mn) phase along the [111] direction of the crystal.

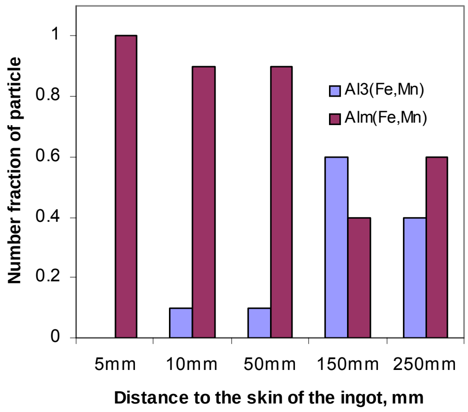

Figure 3: Relative amount of different iron bearing particles through the thickness of the ingot.

Figure 3 shows the relative amount of different constituent phases as a function of distance to the skin of the ingot. As can be seen, the relative amount of different constituent particles is dependent upon the local cooling rate in the ingot. In the samples close to the skin of the ingot, Al_m(Fe,Mn) is the predominant iron-bearing particle, where Al_3(Fe,Mn) particles can only be found occasionally. However, in the samples with 150 and 200 mm distances to the skin, where the local cooling rate is the lowest in the ingot, the amount of Al_3(Fe,Mn) particles is about the same as Al_m(Fe,Mn) phase. These results suggest that increasing local cooling rate favors the formation of Al_m(Fe,Mn).

3.3 Phase Selection of Intermetallic Iron-bearing Particles

Al_mFe phase can often be found in the grain refined commercial purity AA1xxx(Al-Fe-Si) alloys, in which, $Al_6(Fe,Mn)$ and $Al_3(Fe,Mn)$ phases form as dominant particles. The phase selection in the alloy is strongly dependent on the cooling rate of the ingot and grain refinement history [9,14-16]. It has been found that high solidification rate favors the formation of Al_mFe and $Al_6(Fe,Mn)$ particles. Successful grain refinement with Al-Ti-B or Al-Ti-C has also the influence of promoting the formation of Al_mFe phase because TiB_2 and TiC particles in grain refiners can act as heterogeneous nucleation sites for Al_mFe particle [16]. The influence of solidification rate on the phase selection of iron-bearing particles has been attributed to the influence of growth velocity V of the eutectic phase on the growth temperature T_G of different intermetallic eutectic phases [14-16].

$$T_G=T_{EU}-kV^{1/2}$$

Where, T_{EU} is the equilibrium eutectic temperature of intermetallic particle and k is a constant. When different intermetallic eutectic phases compete in the same system, the eutectic that has higher growth temperature will predominate. It can be expected that the growth velocity of the eutectics increase with increasing solidification rate of the alloy. The variation of the relative fraction of the $Al_m(Fe,Mn)$ phase in the present 5182 alloy with local cooling rate in the ingot can also be explained by this equation. Under high local cooling rate, the $Al_m(Fe,Mn)$ phase has higher growth temperature than the $Al_3(Fe,Mn)$ phase and, therefore predominates in the ingot.

However, contrary to the phase selection in AA1xxx alloys, no local area with $Al_6(Fe,Mn)$ or $Al_6(Fe,Mn)+Al_3(Fe,Mn)$ as predominant iron-bearing particles has been found in the present ingot. The absence of $Al_6(Fe, Mn)$ in the present 5182 alloy must be due to the composition difference between AA5182 alloy, which has a high concentration of Mg and 0.22 wt.% Mn, and AA1xxx alloys. It has been found that a low concentration (0.04 wt.%) of Mn and Cr can suppress the formation of Al_6Fe and promote the formation of Al_mFe in a wide freezing rate range in an AA5005 alloy containing, in wt%, Si 0.10, Fe 0.45, Cu 0.11 and Mg 0.69 [17]. Maggs found that a trace Mg concentration in an Al-4%Fe-0.2%Si alloy had the influence of extending the solidification velocity range for the formation of Al_3Fe phase [18]. It suggests that Mg has the influence of promoting the formation of Al_3Fe over Al_6Fe. So the suppression of the formation of $Al_6(Fe,Mn)$ in the present alloy could be due to the Mg and Mn contents.

3.4 A Eutectoid Phase Transformation from $Al_m(Fe,Mn)$ to $Al_3(Fe,Mn)$

Figure 4 (a) shows the morphology of the primary particles after 7h of preheat treatment at 520 °C. No significant change can be observed in the morphology of the iron-bearing primary particles except for some local spheroidization. However, when the particles are examined in FEG-SEM at higher magnifications, a significant morphology change for the iron-bearing particles can be observed. About 90%, in area fraction, of the iron-bearing particles have changed into a lamellar structure distributed with many dark spots, as shown in Figure 4(b). The dark spots on the transformed particles have been identified to be pure aluminium by EDX. The area fraction of the Al spots on the particles has been measured for ten particles. The average area fraction is about 24.5%. The composition of the transformed iron-bearing particles together with the Al spots measured by electron micro-probe shows about the same composition as the as-cast $Al_m(Fe,Mn)$ particles, implying that the $Al_m(Fe,Mn)$ particles may have transformed into $Al_3(Fe,Mn)$ and Al phase. The area fraction of the transformed iron-bearing particles is very close to the fraction of $Al_m(Fe,Mn)$ phase in the as-cast iron-bearing particles, indicating that a phase transformation has occurred on most of the $Al_m(Fe,Mn)$ particles.

This phase transformation has been confirmed by SADP in TEM and EDX composition measurement of the intermetallic branches, which have been identified to be $Al_3(Fe,Mn)$ phase, in the transformed particles.The phase transformation can be expressed as
$Al_m(Fe,Mn) \rightarrow Al_3(Fe,Mn) + (m-3)Al$.

The phase transformation is a eutectoid reaction. It can be calculated that, during transformation, about 23-28 vol.% Al is excluded from the particle, provided m=4.0-4.4. This is in agreement with the measured area fraction of Al phases in the transformed particles.

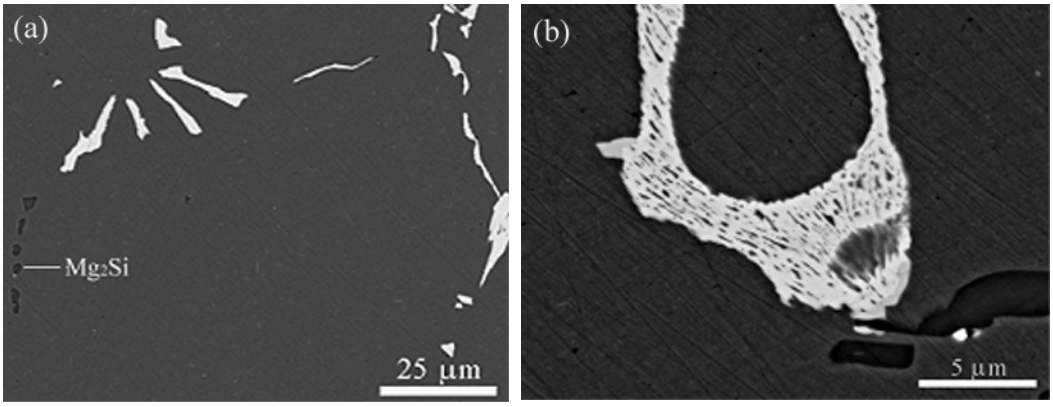

Figure 4: FEG-SEM image of primary particles after homogenization at 520 °C for 7h

An isothermal heat treatment has been conducted to study the kinetics of the phase transformation. The phase transformation fraction as a function of heating time is shown in Figure 5. As can be seen, the phase transformation can be completed in a short time during heating at 520 °C. During heating at 470°C, the phase transformation rate is much lower and the phase transformation needs a long incubation time. It is well known that the coarse intermetallic particles in 5xxx alloy are undesirable for the final sheet forming and stamping operations.

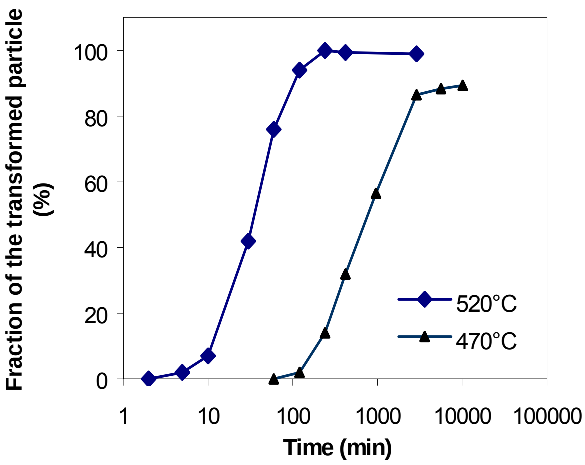

Figure 5: Overall fractional transformation rate of the eutectoid phase transformation during isothermal treatment at 470 and 520 ºC.

Since the phase transformation can change the coarse skeletal shaped $Al_m(Fe,Mn)$ particles into lamellar shaped $Al_3(Fe,Mn)$ particles, it can be expected that the transformed particles are easier to be broken up during hot rolling process. Actually, this has been confirmed by a recent research work by Baldacci et al [19]. They have found that the $Al_m(Fe,Mn)$ particles in the preheat-treated 5182 alloy are much easier to be broken up and spheroidized than the particles in the as-cast alloy without preheat treatment.

4. Conclusions

The dominant iron bearing intermetallic particles have been identified to be $Al_m(Fe,Mn)$ and $Al_3(Fe,Mn)$ instead of $Al_3(Fe,Mn)$ and $Al_6(Fe,Mn)$ in a commercial DC-cast AA5182 alloy. In the ingot, the fraction of the $Al_m(Fe,Mn)$ particles increases with increasing local cooling rate. During preheat treatment, a eutectoid phase transformation $Al_m(Fe,Mn) \rightarrow Al_3(Fe,Mn) + Al$, occurs in the alloy. The $Al_3(Fe,Mn)$ particles transformed from $Al_m(Fe,Mn)$ has many aluminium flakes and spots on their body. At higher temperature, the phase transformation can be finished in a relatively short time; at low temperature, the transformation needs a very long time. The phase transformation of iron bearing particles can be favorable for the formability of AA5182 alloy.

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