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PRECIPITATES IN LONG TERM AGING Al 5083 ALLOY

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Abstract

Al 5083 alloy aged at 343 K (70 ℃) for 3-30 months was evaluated by high resolution transmission electron microscopy (HRTEM) results show that both thickness and continuity of β phase formed at grain boundaries increase with time. GP zones are observed in Al 5083 aged at 343 K for 3 months for the first time. TEM results reveal that GP zones can form homogeneously with the help of vacancies as well as heterogeneously at dislocations near grain boundaries, and the mechanism are explained in detail. Mg_2Si precipitate was found to form at the grain boundary and energy-dispersive X-ray spectroscopy (EDS) results reveal that a Mg, Si (atomic ratio 1:1) rich phase nucleates at the interface of Mg_2Si-matrix. β' phase(Al_3Mg_2, hexagonal) is observed to form at grain boundaries of Al 5083 alloy aged at 343 K for 30 months. β phase was found at the grain boundary of Al 5083 aged at 343 K for 12 months using EDS.

Introduction

AA 5083 alloys are important commercial alloys for their application in transportation industries [1] as well as in marine structures[2]. They have high specific strength, excellent ductility, good corrosion resistance in marine environments as well as their weldability[3]. However, in Al 5083 alloys, Mg is supersaturated and will precipitate at dislocations and grain boundaries when exposed to elevated temperature for a long time[4]. Precipitation (mainly β phase, Al_3Mg_2, fcc) formed at grain boundaries is directly responsible for the intergranular corrosion (IGC) and stress corrosion cracking (SCC) behavior of these alloys [5]. In previous studies [6,7], differential scanning calorimetry (DSC), resistivity measurements, and TEM were used to investigate the precipitation behavior in Al-Mg alloy, and the precipitation sequence [8] in Al-Mg alloys was found to be as follow:

Supersaturated solid solution(α)→GP zones→β''→β'→β(Al_3Mg_2)

Recently, several TEM studies have been done to characterize the precipitates formed in Al 5083 alloy aged at elevated temperature (>343K). Searles *et al.*[9] aged their samples at 343K for up to 333 hours, and their results revealed that β phase formed heterogeneously at grain boundaries. When the aging time is as long as 198 hours, the grain boundaries were fully covered by β phase, and then β phase appeared to be discontinuous when aging time is longer than 262 hours. Goswami *et al.*[10] studied the precipitates in Al 5083 alloy aged at 448K in detail. Their research showed that, discrete β phase formed at grain boundaries after aging at 448 K for 1 hour, and when the aging time increased to 240 hours, β phase became continuous. Zhu. *et al.*[11] found that, β phase formed at grain boundaries as well as on preexisting Mn-Fe-Cr particles when Al 5083-H131 alloy was aged at 448 K for 15 days.
Even though a wealth of research has been done on the precipitates in Al 5083 alloys, these studies are mainly conducted at relatively high temperature for a short time (less than one month). The aim of this work is to characterize precipitates formed in Al 5083 alloys aged at low temperature (343 K) for a long time (as long as 30 months), which is more relevant to outdoor thermal exposure.

Experimental Procedures

For this study, the Al 5083 alloy, which was received in the H-131 temper condition from Alcoa, was cut into cubes with a side length of 1cm. Subsequently, these samples were aged at 343 K for 3, 12, and 30 months.
TEM was used to characterize the microstructure of 5083-H131 alloys aging at 343 K for different times. The characterization process were performed using FEI Titan S 80 TEM/STEM (200/300 kV) and JEOL 2200FS (200 kV) TEM. EDS quantification was performed using the Cliff-Lorimer quantification routine built into the Bruker Esprit software package. TEM foils were prepared by ion milling techniques.

Results and Discussion

Fig. 1 is a bright field STEM image of the Al 5083-H131 alloy before aging. Four dark particles, one is in rod shape (as highlighted by 1) and the other three (2, 3, and 4) are equiaxed, were found within the grain matrix. Li et al.[12] and Zhu's[11] research shows that particle 1 to 4 are Fe-Cr-Mn enriched with a Al_6Mn type. Moreover, no precipitates were found to form at the grain boundary, as indicated by a series of dark arrows.

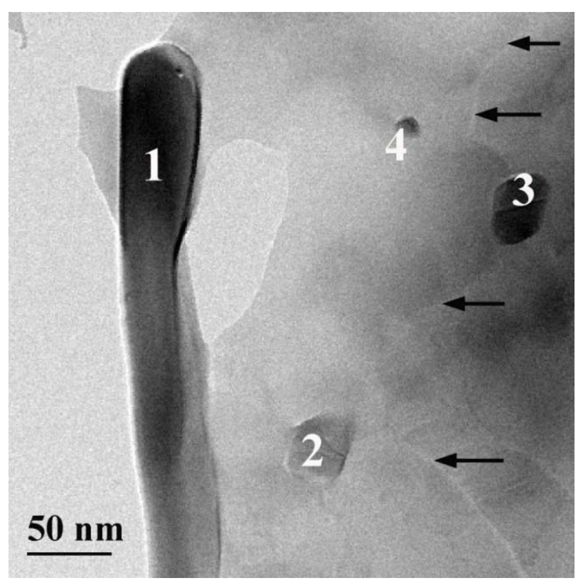

Figure 1. Bright field STEM image of Al 5083-H131 alloy before aging.

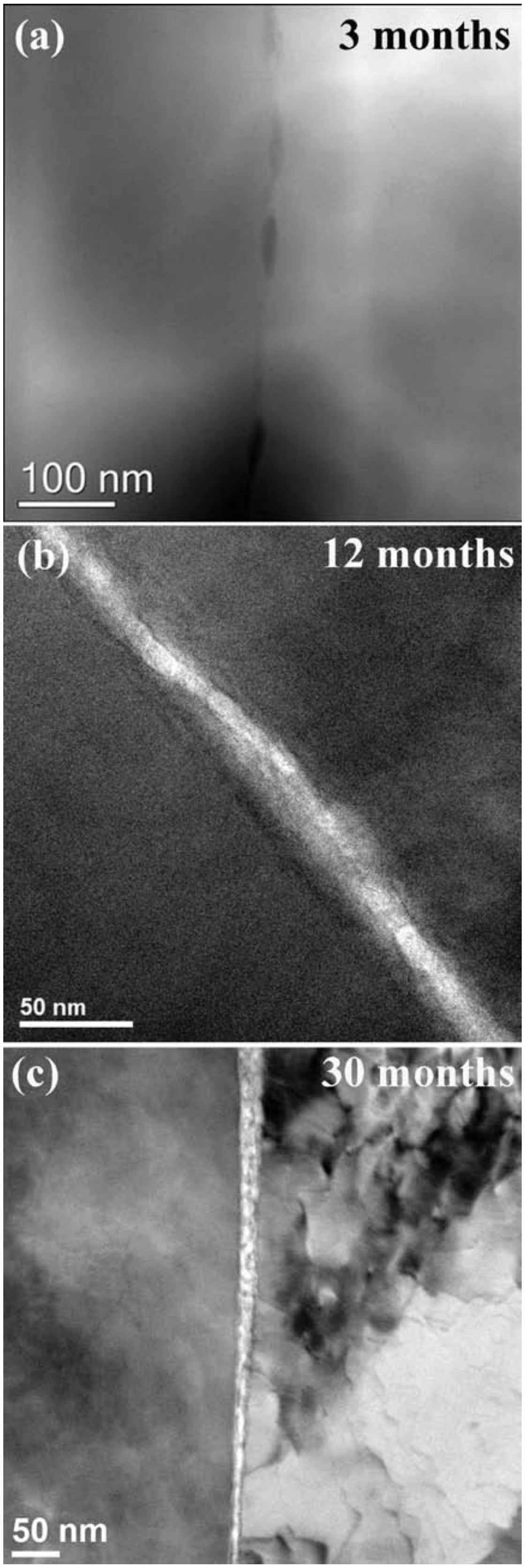

Figure 2. STEM image of precipitates formed at the grain boundaries of Al 5083-H131 alloy aged at 343 K for 3 (a), 12 (b) and 30 (c) months.

Fig. 2 shows three STEM images of precipitates formed at grain boundaries of Al 5083 alloys aged at 343 K for 3, 12 and 30 months. As shown in Fig. 2(a), discrete precipitates formed at the grain boundary of Al 5083 H-131 after aging for 3 months. The average length and thickness of these particles are 65 nm and 12 nm respectively, and the distance between these particles is about 100 nm. Fig. 2(b) is a STEM image of precipitates formed at the grain boundary after aging for 12 months. The thickness of these precipitates increases to 20 nm, and the distance between two adjacent precipitates is less than 10 nm. When aging time is as long as 30 months, grain boundaries are fully covered by precipitates (as shown in Fig. 2(c)). Also, the thickness of beta phase increases to 30 nm. Therefore, both thickness and continuity increase with aging time when Al 5083 alloy samples are exposed at a constant temperature (343 K), which is consistent with the general aging response results of Goswami[13].

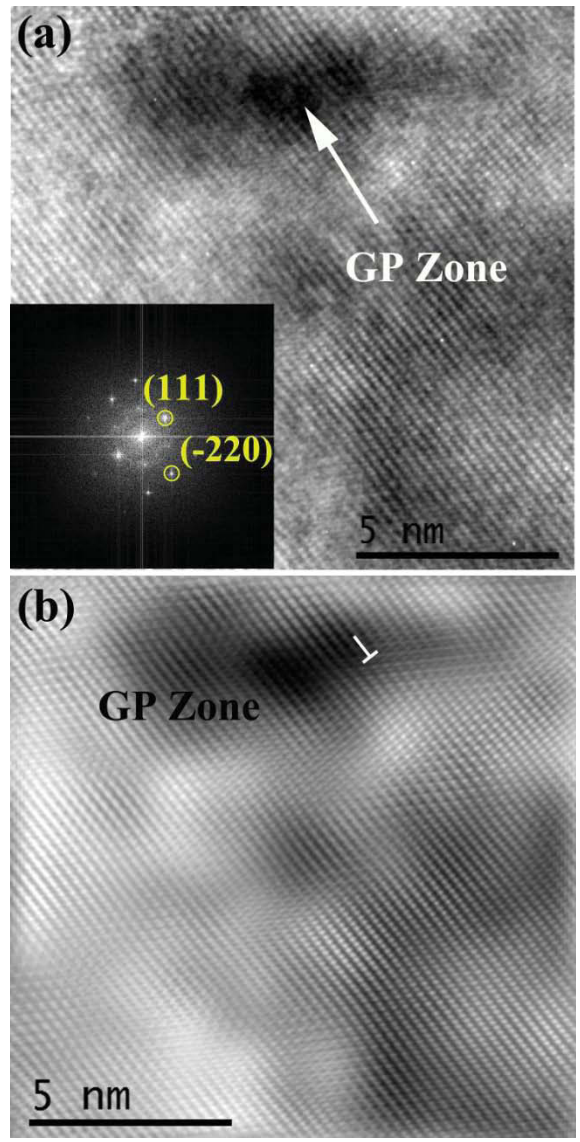

Figure. 3 (a) TEM image of a likely GP zone formed in Al 5083-H131 alloys aged at 343 K for 3 months, and (b) inverse FFT pattern of (a).

Fig. 3(a) shows a possible GP (as highlighted by the white arrow) zone formed within the grain matrix of Al 5083 alloy aged at 343 K for 3 months. The size of the GP zone is about 5 nm in length and 1.8 nm in width. Previous research on GP zones formed in Al-Mg-Si[14,15], Al-Zn-Mg[16,17] and Al-Cu[18] alloys reveals that these zones are small in size (several nm), and tend to cause a white-black contrast in the image due to the coherency misfit strain perpendicular to the zones. The inset Fast Fourier Transform (FFT) pattern of the image shows that, the diffraction spots are only due to Al matrix. Fig. 3 (b) is the corresponding Inverse FFT (IFFT) pattern of Fig. 3(a). As highlighted by the symbol "⊥", an edge dislocation is found within the GP zone. The dislocation could provide a heterogeneous precipitation site for the GP zone, which is consistent with the result of Hu[19]. In addition to heterogeneous nucleation at dislocations, GP zones are also able to nucleate homogeneously, as found in Al-Cu alloy[20]. However, during the homogeneous nucleation process associated with GP zone, excess vacancies play an important role[19]. More work is needed to confirm the presence and role of the GP zones in these samples.

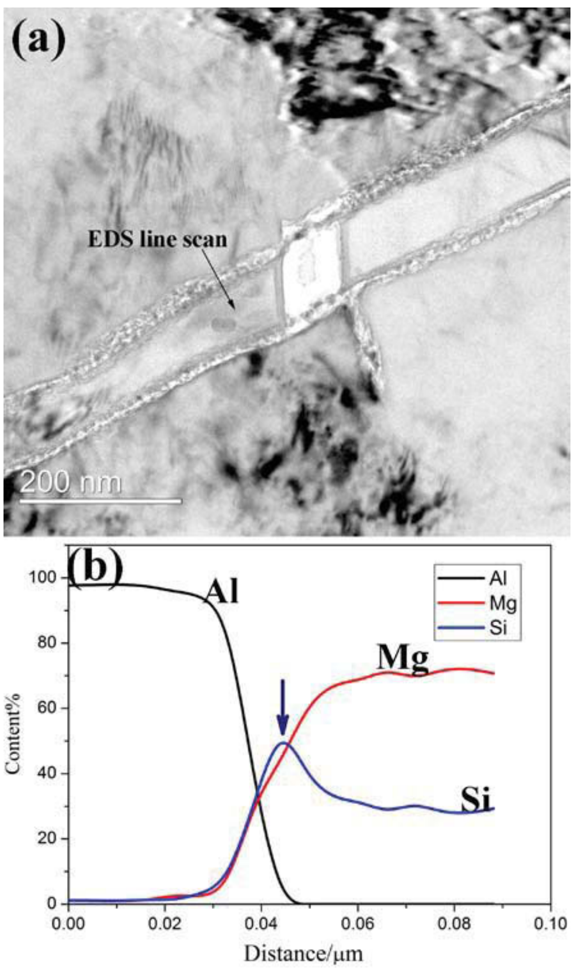

Figure 4. (a) Mg_2Si formed within the grain matrix of Al 5083-H131 aged at 343 K for 30 months, and (b) EDS line scan results across the matrix-particle interface.

Fig. 4 (a) shows a rod shape particle formed within the grain matrix of Al 5083-H131 aged at 343 K for 30 months. Its length is about 800 nm, and its thickness is nearly 100 nm. Moreover, another precipitate was found to form on the matrix-particle interface, and the thickness of this phase is about 20 nm. Fig. 4(b) is the EDS line scan result across the Al matrix- particle interface, as marked by a dark arrow in Fig. 4(a). EDS line scan results reveal that, for the rod shaped particle, the main components are Mg and Si, and the atomic ratio of these two elements is about 2:1, which indicates that this particle is Mg_2Si. Actually, Mg_2Si precipitate in Al 5083 alloy has already been observed and thoroughly investigated by previous researchers and their results shows that Mg_2Si is a kind of pre-existing particle. The EDS line scan results also shows that the precipitate formed at the matrix-particle interface is mainly Mg and Si as well, but their ratio is approximately 1:1(as indicated by the arrow in Fig. 4(b)). This finding shows that, even though Mg_2Si and Al_6Mn type Fe-Cr-Mn rich particles are both pre-existing particles formed during the solidification process of Al 5083 alloy, precipitates formed on these particles during the aging process can be totally different.

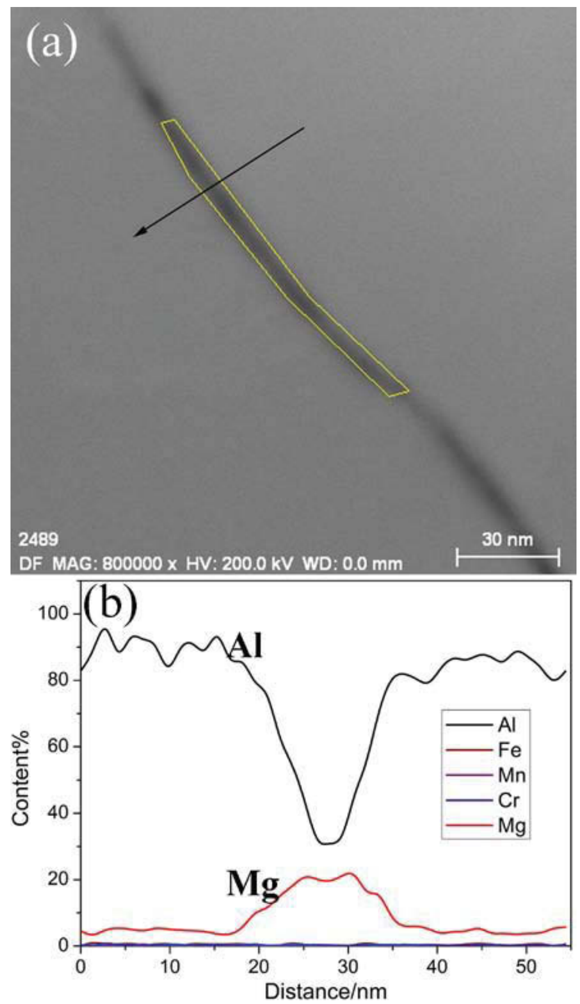

Figure 5. (a) STEM image of β phase (Al_3Mg_2) fromed at the grain boundary of Al 5083-H131 alloy aged at 343 K for 12 months, and (b) EDS line scan results across β phase at grain boundary.

Fig. 5 (a) is dark field STEM image of precipitate formed at the grain boundary of Al 5083 H131 aged at 343 K for 12 months, and the grain boundary is almost fully covered by the precipitate. Fig. 5(b) shows the EDS line scan result of Al and Mg across the grain boundary. Based on EDS result, Al to Mg atomic ratio is about 3:2. To further confirm the result, Al and Mg peaks in EDS spectra collected from the area within the frame in Fig. 5(a) were quantified. The result showed,59 at. pct Al, 41 at. pct Mg, which is the same as the EDS line scan profile.

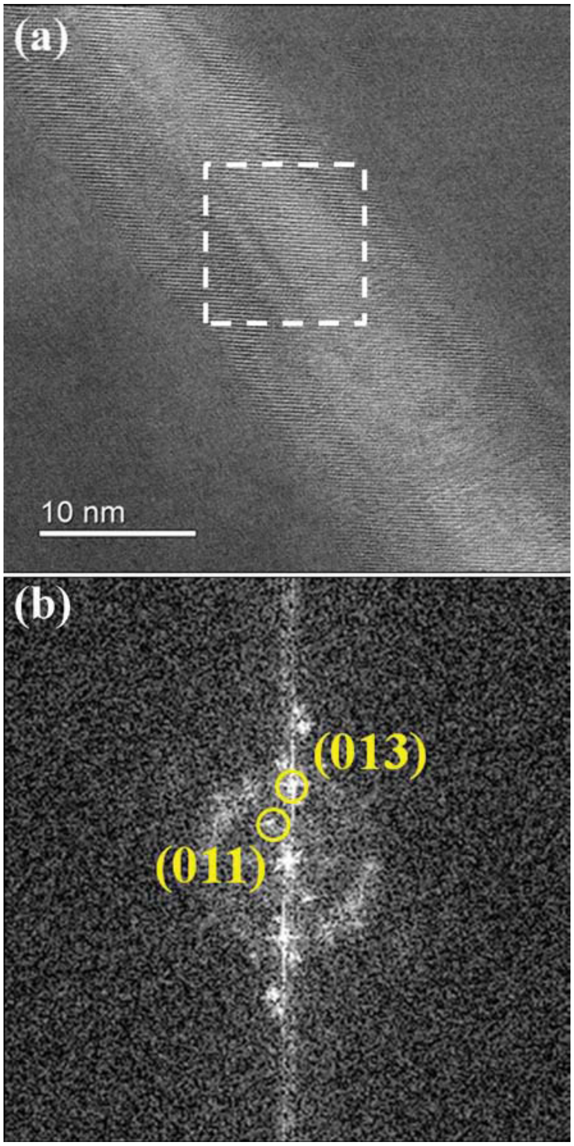

Figure. 6 (a) STEM image of β' phase (Al_3Mg_2, hexagonal) formed at the grain boundary of Al 5083-H131 aged at 343 K for 30 months, and (b) FFT pattern of the dashed frame area in (a).

Fig. 6(a) is a bright field STEM image of precipitate formed at the grain boundary of Al 5083-H131 aged at 343 K for 30 months. The thickness of the precipitate is about 14 nm. Fig. 6(b) is the FFT pattern of the precipitate within the dashed frame. Careful examination of the diffraction spots reveals that this precipitate is Al_3Mg_2 with a hexagonal structure (JCPDF40-0903), and its lattice parameter is a =5.73Å, c=9.54 Å. According to pervious research [21], Al_3Mg_2 precipitates with a hexagonal structure are named as β' phase.

Results from Fig. 5 and Fig. 6 indicate that both β phase and β' phase can form and exist at 343 K, which is different from results reported previously showed that β' phase precipitates between 373 K and 523 K, and then β phase begins to nucleate at around 473 K. Therefore, given enough exposure time, both β phase and β' phase can form at 343 K.

Conclusions

Al 5083-H131 alloy samples were aged at 343 K for 3, 12, and 30 months, and TEM was used to characterize the microstructure of these samples. TEM results reveal that both precipitate thickness and continuity increase with time. What appeared to be a GP zone, 5 nm in length and 1.8 nm in width, was observed in sample aged for 3 months. Edge dislocations are believed to assist the heterogeneous nucleation of GP zones. A rod shaped Mg_2Si particle was found to form within the Al grain matrix, and Mg, Si (1:1 in atomic ratio) rich phase precipitates at the interface of the matrix and the rod shaped particle. β' phase (Al_3Mg_2, hexagonal) was observed in Al 5083 alloys aged at 343K for 30 months, and β phase (Al_3Mg_2, fcc) was shown to exist in Al 5083 alloy aged at 343K for 12 months by EDS.

Acknowledgements

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