

Atom Probe Tomography Characterisation of Solute Clustering in AA6111

Sha. Li^{1,2}, Ross K.W. Marceau^{1,2}, Gang Sha^{1,2}, Warren J. Poole³ and Simon P. Ringer^{1,2}

¹ARC Centre of Excellence for Design in Light Metals

²Australian Centre for Microscopy & Microanalysis, The University of Sydney, NSW, 2006, Australia

³Department of Materials Engineering, The University of British Columbia, Vancouver, BC, V6T 1Z4,
Canada

A series of ageing treatments have been performed on AA6111 alloy samples for various periods ranging from 4 mins to 2 weeks ageing between room temperature and 180 °C. The microstructures of these alloy samples have been carefully characterised using atom probe tomography in order to obtain quantitative information about the solute clusters and early-stage precipitates that are formed. Intriguingly, an iso- yield strength regime has been measured for samples aged variously with respect to temperature and duration, generating different precipitate microstructures. Ageing at a higher temperature produced larger early-stage precipitates at lower number density. In contrast, the yield strength of naturally aged samples increased with ageing time at room temperature and initially produced a higher number density of smaller solute clusters followed by a development in the microstructure to a lower number density of early-stage GP zone precipitates. The strengthening effect of different precipitate microstructures formed under different ageing treatments has been addressed.

Keywords: AA6111, atom probe tomography, solute clusters, precipitation hardening.

1. Introduction

The 6111 Al alloy is a precipitation hardened alloy, and has been considered as potential candidate materials for replacing steel components used in automobiles in order to considerably reduce vehicle weight [1-7]. The microstructural evolution of the alloy during natural and artificial ageing has drawn significant amount of research interest in recent years [1-8]. Previous studies have focused on the relationship between microstructure and mechanical properties and revealed the formation of solute clusters prior to the precipitation of the strengthening β'' phase. However, less work has been done to develop thorough understanding of the microstructure formed during early-stage ageing treatments. The chemical composition and characteristics of solute clusters are not well defined since they are generally hard to resolve using conventional transmission electron microscopy (TEM). Atom probe tomography (APT) is a useful microanalysis method to provide the atomic resolution and ability to identify small solute enriched features precisely. In this research, APT has been employed to characterise solute clusters and early-stage precipitates formed in AA6111 alloy.

2. Experimental procedure

A commercial aluminium alloy 6111 was used for this investigation having a composition 0.75 wt.% Mg, 0.75 wt.% Cu and 0.63 wt.% Si (the balance Al). Square-sectioned samples (0.5×0.5 mm and 10 mm in length) were solution heat treated in a salt bath for 10 mins at 560 °C followed by water quenching. Three samples were subsequently artificially aged in an oil bath at different temperatures and ageing times. The remaining four samples were naturally aged at room temperature for increasing periods of time. All the ageing conditions are listed in Table 1. Mechanical properties were determined from standard tensile testing.

The APT work was performed on sharp needles with tip radii ~50-100 nm prepared by standard two-stage electropolishing techniques using a Local Electrode Atom Probe (LEAP) instrument at

~ 20 K with a pulse fraction (the ratio of the pulse voltage to current standing voltage) of 20% and a pulse repetition rate of 200 kHz, under a vacuum of below 10^{-10} Torr. Identification of precipitates and solute clusters was carried out using a combination of the maximum separation [9] and Core-Linkage [10] cluster-finding algorithms in a manner similar to that reported in [11].

Table 1: Ageing conditions of AA6111 samples

Natural ageing	Artificial ageing
Room temperature 4.8×10^2 mins (8 hours)	90 °C for 1.44×10^3 mins (24 hours)
Room temperature 6.06×10^3 mins (4 days 5 hours)	150 °C for 20 mins
Room temperature 1.01×10^4 mins (1 week)	180 °C for 4 mins
Room temperature 2.02×10^4 mins (2 weeks)	

3. Results and Discussion

3.1 Mechanical properties

Results of the tensile testing of both the naturally aged samples and artificially aged samples are shown in Fig. 1a and 1b respectively; given as yield strength as a function of ageing condition. It can be seen that there is a monotonic increase in the yield strength with increased natural ageing time, whilst the samples artificially aged for various conditions reach a similar iso-yield strength level. The aim of this work was to investigate the effect of ageing time and temperature on the microstructural development of this alloy to understand the difference in behaviour observed in Figs. 1a and 1b.

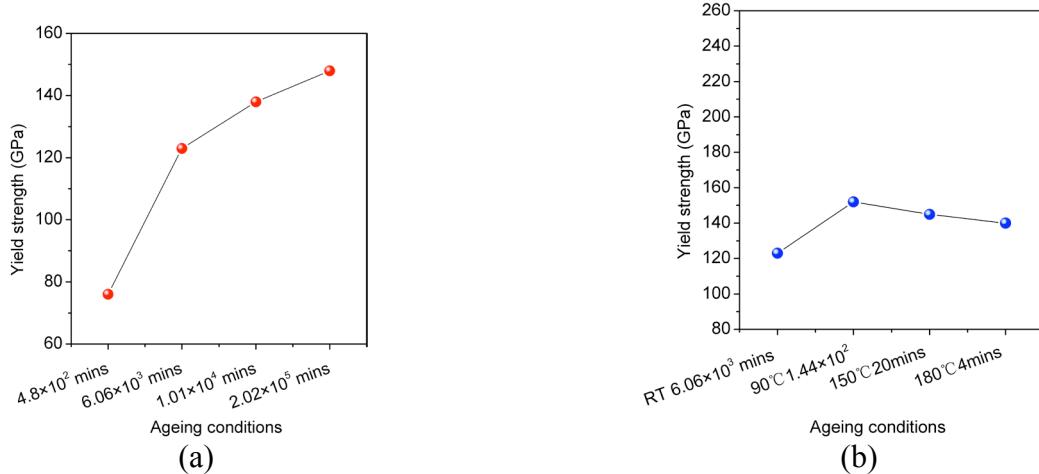


Fig. 1: The yield strength of AA6111 samples aged (a) at room temperature for different ageing time (naturally aged samples) and (b) artificially aged samples and also a sample aged at room temperature for 6.06×10^3 mins (iso-yield strength samples).

The contribution to the yield stress from precipitation hardening, σ_{ppt} , is related to the relevant microstructural variables including the size, chemical composition and volume fraction of the

features. The volume fraction can be evaluated by characterising the size and number density of such features.

3.2 Microstructure of naturally aged AA6111 samples

Fig. 2 shows three-dimensional (3D) reconstructions of the atom probe data for the naturally aged AA6111 samples. Localised solute enriched regions can be observed in all four conditions. As the ageing time at room temperature increases, the clustering becomes more obvious as these regions grow larger and appear denser.

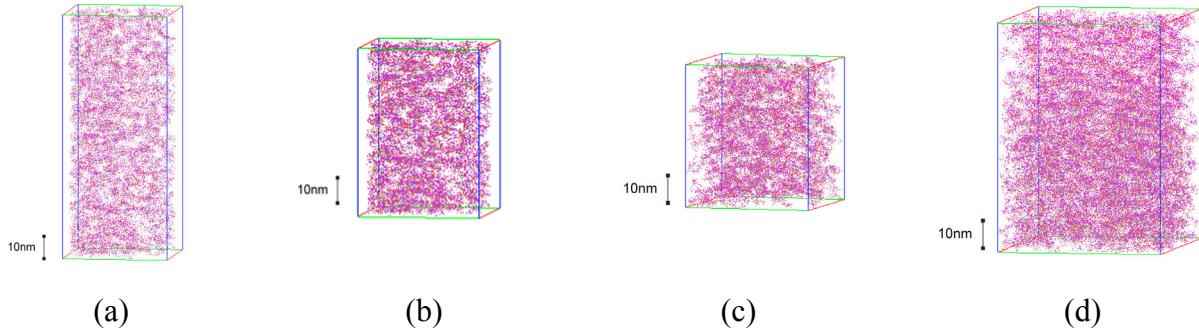


Fig. 2: 3D solute atom maps of clustered solute atom (Mg+Cu+Si) features in samples aged at room temperature for (a) 4.8×10^2 mins (b) 6.06×10^3 mins (c) 1.01×10^4 mins (d) 2.02×10^4 mins.

Given that changes in the microstructure can be observed visually in the APT data, the number density results presented in Fig. 3 from application of cluster-finding algorithms have been limited to features ≥ 30 solute atoms in size.

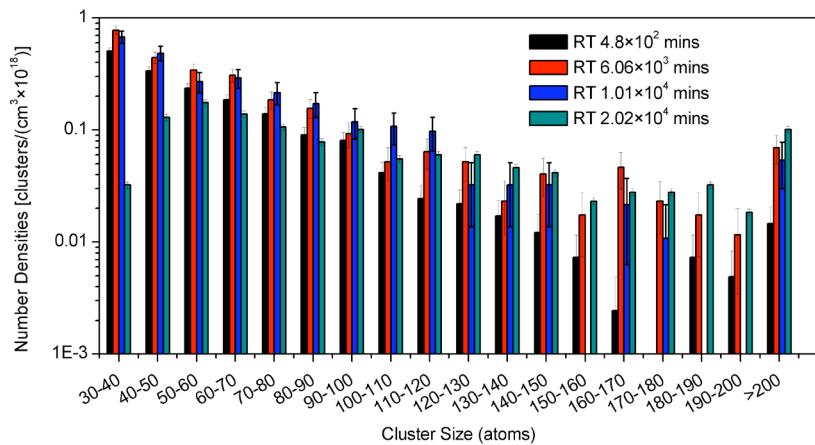


Fig. 3: Number density versus size (number of Mg+Cu+Si solute atoms) of naturally aged AA6111 samples.

The trend in number density is more easily interpreted when plotted as mean number density for each naturally aged condition, as shown in Fig. 4a. At this stage attention is also drawn to Fig. 4b, which shows the mean size of the solute atom (Mg+Cu+Si) features identified by cluster-finding analysis. The mean number density of these solute features increases initially, as does their mean size, which correlates to the increase in yield strength observed in Fig. 1a. However, after 2 weeks (2.02×10^4 mins) of natural ageing there is a decrease in the number density and corresponding increase in size of these solute rich features. This trend is concomitant with a continued increase in the yield strength of the AA6111 samples during natural ageing (Fig. 1a). Thus, there is critical

development in the microstructural evolution at this time and; according to both the size information in Fig. 3 and Fig. 4b, and the equiaxed morphology of the features observed in Fig. 2d; is most likely due to the formation of pre- β'' precipitates. Previous research [2,8] indicates that these could be either solute clusters or, in the case of larger features; spherical Guinier-Preston (GP) zones that grow larger with ageing.

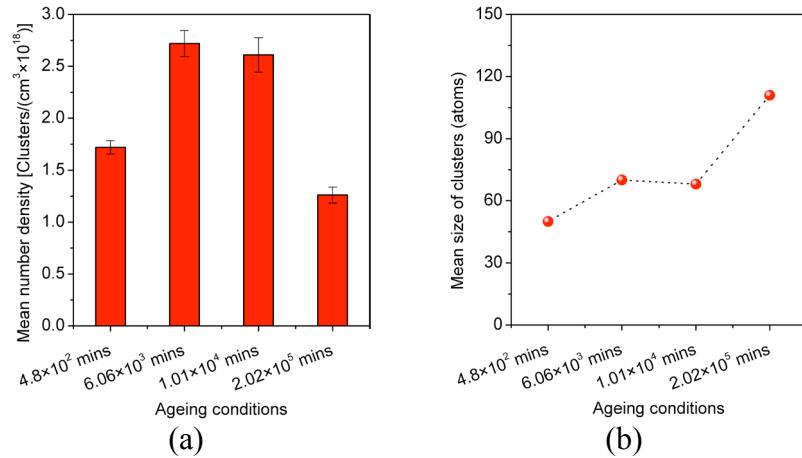


Fig. 4: (a) Mean number densities and (b) mean size of (Mg+Cu+Si) features from naturally aged AA6111 samples.

3.3 Microstructure iso-yield strength AA6111 samples

Fig. 5 shows 3D reconstructions of the atom probe data for the AA6111 samples with iso-yield strength, having been aged at different conditions. Again, there are localised solute enriched regions in the data with most of these having a roughly spherical shape; however some of the larger features have a slightly elongated rod shape.

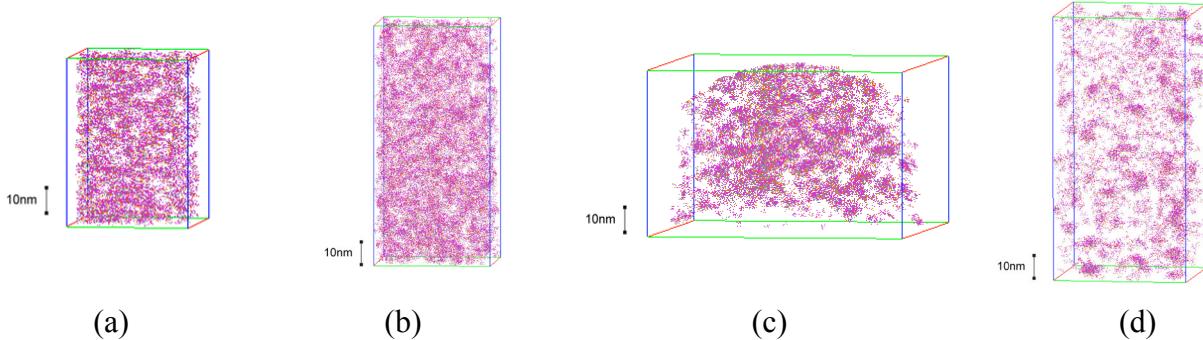


Fig. 5: 3D atom maps of clustered solute atom (Mg+Cu+Si) features in AA6111 samples aged at (a) RT for 6.06×10^3 mins (b) 90°C for 1.44×10^3 mins (c) 150°C for 20 mins (d) 180°C for 4 mins.

Results for number density versus size of clustered features containing Mg+Cu+Si solute atoms in the samples belonging to the iso-yield strength group are displayed in Fig. 6. The mean number density and size of these features are summarised in Figs. 7a and 7b respectively. From Fig. 7a, it can be seen that the mean number density of solute atom features in the iso-yield strength samples decreases significantly with increasing ageing temperature. On the other hand, the mean size of these features increases (Fig. 7b). Thus, there is a balance between size and number density that corresponds with the iso-yield strength level observed in Fig. 4b, indicating that the volume fraction of solute-rich features in the microstructure plays a key part in the level of strengthening. When

compared to the naturally aged samples, the artificially aged samples have a larger mean size of solute-rich features (Fig. 4b and Fig. 7b) and a correspondingly higher level of yield strength (Fig. 1a and 1b). As mentioned earlier, the morphology of these features is mostly spherical with the largest having a slightly elongated form; indicating that a high percentage of the particles present are GP zone precipitates but the beginning of the formation of needle-like β'' precipitates is also evident.

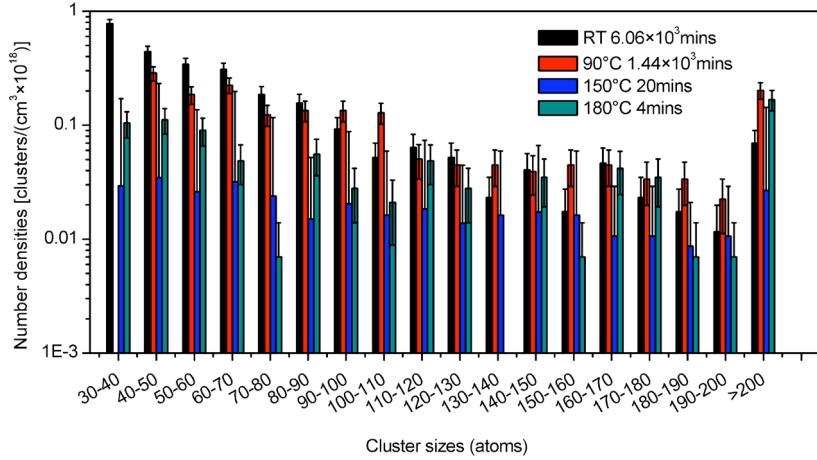


Fig. 6: Number density versus size (number of Mg+Cu+Si solute atoms) of AA6111 samples aged at different conditions.

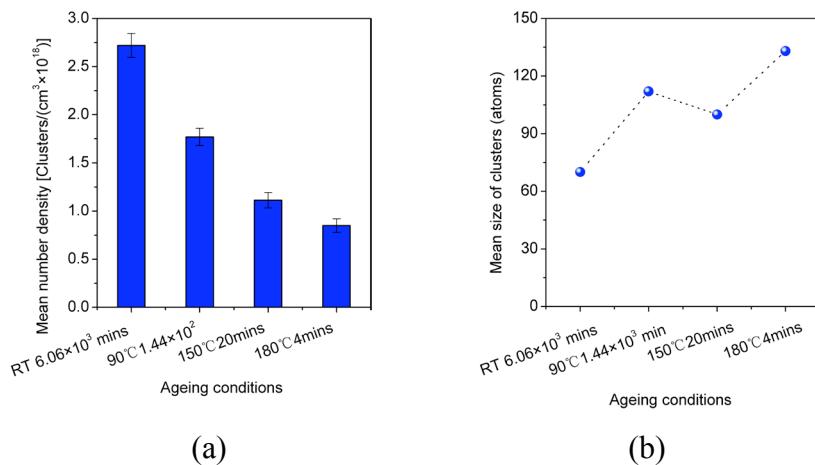


Fig. 7: (a) Mean number densities and (b) mean size of (Mg+Cu+Si) features from the iso-yield strength aged AA6111 samples.

4. Conclusions

Atom probe tomography, employing sophisticated cluster-finding analysis techniques, has enabled identification, reconstruction and quantitative analysis of the change in distribution of solute clusters and early stage precipitates in two groups of AA6111 samples with different ageing conditions.

In the group of samples naturally aged at room temperature for increasing time, the increase in yield strength is attributed to a high number density of solute atom (Mg+Si+Cu) clusters, which increases with ageing. However, as natural ageing continues the number density of these types of

features decreases corresponding with an increase in the mean size. The monotonic increase in yield strength is sustained by the development of larger GP zone precipitates.

For the group of samples that displayed a similar level of yield strength (the iso-yield strength group) despite various levels of both natural and artificial ageing; there was a decrease in the number density of solute-rich features concurrent with an increase in their mean size. As a result, the effectively constant volume fraction of solute-rich features was the dominant factor influencing the trend in mechanical properties observed. The APT results support the literature, which states that spherical GP zones precede the formation of elongated β'' precipitates.

Acknowledgements

This work is supported by the Australian Research Council (ARC) through the ARC Centre of Excellence for Design in Light Metals. The authors are also grateful for scientific and technical input and support from the Australian Microscopy & Microanalysis Research Facility (AMMRF) node at The University of Sydney.

References

- [1] S. Esmaeili, D. Vaumousse, M.W. Zanderbergen, W.J. Poole, A. Cerezo and D.J. Lloyd: Phil. Mag. 87 (2007) 3797-3816.
- [2] X. Wang, S. Esmaeili and D.J. Lloyd: Metall. Mater. Trans. A 37 (2006) 2691-2699.
- [3] S. Esmaeili, D.J. Lloyd and W.J. Poole: Acta Mater. 51 (2003) 3467-3481.
- [4] S. Esmaeili, X. Wang, D.J. Lloyd and W.J. Poole: Metall. Mater. Trans. A 34 (2003) 751-763.
- [5] X. Wang, J.D. Embury, W.J. Poole, S. Esmaeili and D.J. Lloyd: Metall. Mater. Trans. A 34 (2003) 2913-2924.
- [6] G.C. Weatherly, A. Perovic, D.D. Perovic, N.K. Mukhopadhyay and D.J. Lloyd: Metall. Mater. Trans. A 32 (2001) 213-218.
- [7] A. Perovic, D.D. Perovic, G.C. Weatherly and D.J. Lloyd: Scripta Mater. 41 (1999) 703-708.
- [8] G.A. Edwards, K. Stiller, G.L. Dunlop, M.J. Couper: Acta Mater. 46 (1998) 3893-3904.
- [9] M.K. Miller and E.A. Kenik: Micros. Microanal. 10 (2004) 336-341.
- [10] L.T. Stephenson, M.P. Moody, P.V. Liddicoat and S.P. Ringer: Micros. Mircoanal. 13 (2007) 448-463.
- [11] R.K.W. Marceau, S.P. Ringer, J. da Costa Teixeira and C.R. Hutchinson: elsewhere in Proceedings of the 12th International Conference on Aluminium Alloys.