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TEM Analysis of an Interrupted Aged 6061 Aluminium Alloy

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Abstract

A novel ageing treatment has been developed in which a conventional, single stage T6 temper is interrupted by a period at reduced temperature (eg. 25-65ºC). This so-called T6I6 treatment has been applied to the Al-Mg-Si alloy 6061 and found to cause simultaneous increases in tensile properties, hardness, and damage tolerance as compared with 6061 T6. Microstructural development in 6061 during ageing to the T6 and T6I6 tempers has been studied by transmission electron microscopy which has revealed that interrupted ageing results in the formation of finer and more densely dispersed precipitates within the alloy.

1. Introduction

The Al-Mg-Si alloys are amongst the most widely used wrought heat treatable aluminium alloys due to their favourable combination of mechanical properties, formability and resistance to stress-corrosion cracking. Ageing behaviour and the precipitation sequence in these alloys is known to be complex. However, it has been widely accepted that the decomposition of supersaturated Al-Mg-Si alloys occurs in the following sequence [1,2]:

Discrete clusters of Mg and Si → co-clusters containing Mg and Si → GP zones → β” precipitates→ β’ precipitates → β (Mg_2Si)

The most effective hardening phase is the needle-like β” [3-5]. The high strength of these alloys is most commonly achieved by subjecting them to a T6 heat treatment performed in the temperature range from 160 to 180˚C, typically for 6-20 hours after solution treatment.

However, recent observations made on a range of aluminium alloys indicate that a modification of the conventional T6 heat treatment, which involves insertion of low temperature ageing stage, may improve mechanical properties of these alloys [6]. The potential for this improvement lies in the control of secondary precipitation that may take place even in some fully aged alloys when exposed to reduced temperatures for extended periods of time. In Al-Li alloys and some magnesium alloys this phenomenon causes reduction in ductility and fracture toughness, which has been ascribed to secondary precipitation of a fine dispersion of the hardening phase throughout the matrix [7]. In Al-Cu-Mg alloys observations made using positron annihilation lifetime spectroscopy (PALS) have revealed that vacancies retained in the alloy after ageing at 180˚C are mobile at lower temperatures [8,9]. In an Al-Cu-Mg-Ag alloy, secondary precipitation has been observed to affect creep performance and manipulation of this phenomenon has been used to improve the creep behaviour of these alloys [10]. The ability to control secondary precipitation has led to the development of a new series of heat treatments designated T6I6 indicating that the standard T6 treatment is interrupted by a dwell period (I) at a lower temperature before resuming the final ageing at the temperature of the initial T6 treatment, or at a slightly different elevated temperature [11]. Investigation of the T6I6 treatment has already been performed on a wide range of aluminium alloys and has proven that this ageing cycle may produce substantial improvements in the mechanical properties of many alloys [6]. The present work was concentrated on understanding the relationship between the microstructure and mechanical properties produced by application of a T6I6 ageing treatment to the commercial alloy 6061.

2. Experimental

The commercial alloy AA6061, whose composition is given in Table 1, was supplied by Comalco in the form of homogenized billet.

Table 1: Composition of the AA6061 in wt. %

Alloy	Si	Mg	Cu	Fe	Cr	Zn	Mn	Ti
AA6061	0.59	0.99	0.25	0.16	0.112	0.002	0.13	0.012

The alloy was solutionized at 560˚C for 2 h in an air-circulating furnace and quenched in cold water. Artificial ageing was performed in an oil bath, followed by quenching in petroleum ether. Three different artificial ageing treatments were applied after the quench for examination. These were: (a) T6 heat treatment by ageing at 177˚C; (b) T6I6/177, achieved by ageing for 20 minutes at 177°C followed by a quench into petroleum ether, ageing at 65°C for 14 days, then re-ageing at 177°C and (c) T6I6/150, achieved by ageing for 20 minutes at 177°C, followed by a quench into petroleum ether, ageing at 65°C for 14 days, then re-ageing at 150°C. Subjecting the T6I6 variants to 20 minutes artificial ageing at 177˚C produced 80% of T6 temper hardness value.

The age hardening behaviour of the alloy subjected to the three heat treatments was monitored by hardness measurements. Mechanical properties in the peak aged conditions were determined for the material prepared in each of the three tempers. Tensile properties were evaluated in accordance with Australian standard AS 1391-1991 and conducted on five samples for each condition. Damage tolerance of the alloy was determined using the chevron notch procedure given in ASTM E1304-97 and conducted for three samples for each condition. It should be noted that the plane strain condition could not be achieved with material used in this study because of its high ductility. Transmission electron microscopy (TEM) was used to study the effect of interrupted ageing on the precipitation processes. Thin foils for TEM were prepared from aged material by conventional techniques and examined in a Phillips CM200 TEM operated at 200 kV.

3. Results and Discussion

3.1 Hardness Measurements and Mechanical Properties

Hardness curves of the alloy specimens obtained under the three heat treatments examined are presented in Figure 1. For the T6 heat treatment at 177˚C, between 15 to 18 hours of ageing was required to reach peak hardness. For the T6I6 treatments, nearly 90% of the T6 peak hardness was achieved after the interrupt stage of 2 weeks at 65°C. The hardness increment occurring at this lower temperature is indicated by the vertical dashed lines in Figure1 a) and b), and is attributed to secondary precipitation. After the interrupt treatments, the T6I6 treatments were resumed by ageing at 177˚C (Figure 1a) or at 150˚C (Figure 1b). For each case examined, the hardness reached a peak following 15 to 18 hours of final re-ageing, and increased the hardness of the material by 7-8% when compared to the T6 temper.

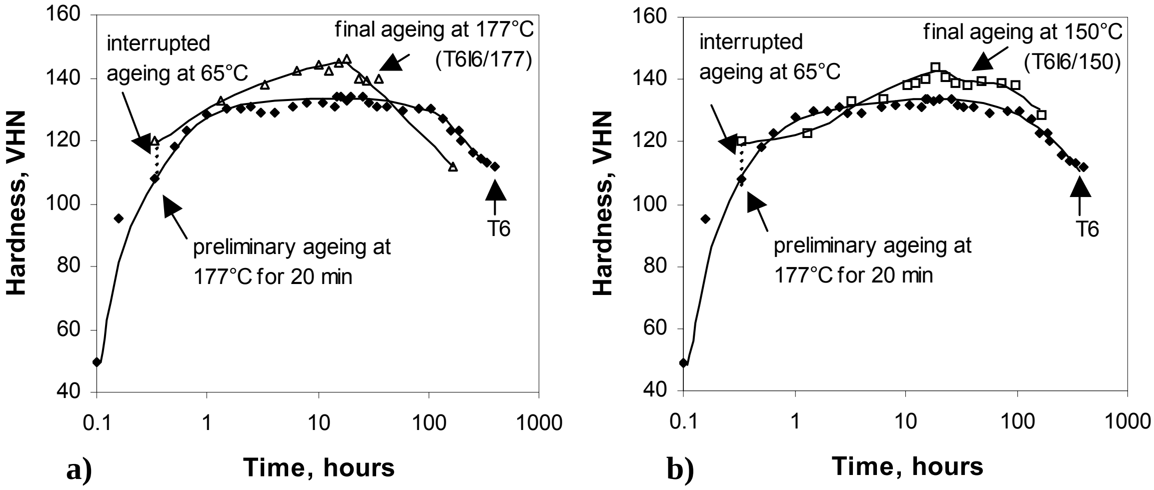

Figure 1: Hardness curves for 6061 for the conventional T6 heat treatment at 177˚C (solid diamonds) and T6I6 treatments examined. Final stage ageing was conducted at a) 177˚C (open triangles, T6I6/177) and b) at 150˚C (open squares, T6I6/150).

A comparison of the tensile properties, as well as the damage tolerance, for the three tempers in the peak aged conditions is given in Figure 2. Figure 2 a) shows that both T6I6 heat treatments produce improvements in the ultimate tensile strength (UTS). A notable improvement in the 0.2% proof stress (~8% higher than that of the T6 temper) was also achieved through the T6I6/177 heat treatment without any significant change to the ductility of the alloy. As evident from Figure 2 b) the most substantial improvement, however, was in the fracture behaviour. The T6I6/177 heat treatment increased the damage tolerance of the alloy by ~ 21%, while in the case of T6I6/150, when a slightly lower re-ageing temperature of 150°C was applied, the alloy was tougher by 36% than in the T6 condition.

It is evident that the T6I6 ageing treatment broadly improves mechanical properties of alloy 6061. In particular, the T6I6/177 heat treatment produces improvements in both the tensile properties and the toughness. If the final stage of this novel heat treatment is conducted at a slightly lower temperature, such as at 150˚C, an even higher improvement in toughness is achieved without significant loss in the tensile properties.

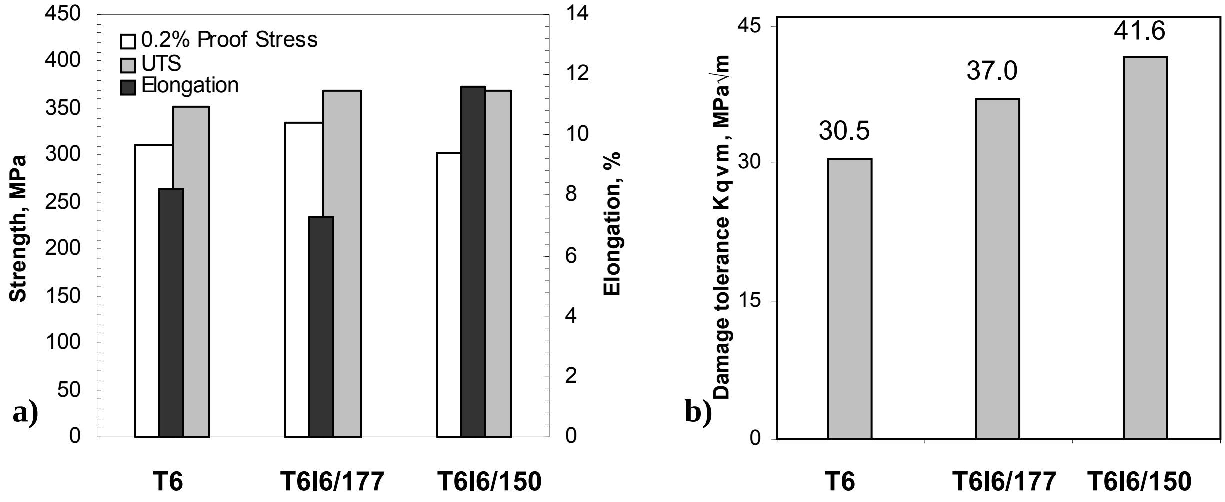

Figure 2: a) Tensile properties and b) damage tolerance of 6061 in T6 and T6I6 peak aged conditions.

3.2 Microstructural Observations

Transmission electron microscopy was performed on each of the three tempers in the underaged, peak-aged and overaged conditions. Figures 3 (a), (b) and (c) show bright field TEM images obtained from the material subjected to the conventional T6 heat treatment. In the specimen aged for 4 hours dark contrast arising from fine precipitates of unresolved shape were observed (Figure 3 (a)). These precipitates were identified as GP zones [1]. A notable amount of needle-like precipitates were also observed to coexist with GP zones. These precipitates, shown also in Figure 3 (b) and aligned in the <001> matrix directions, are identified as β” precipitates [12]. On further ageing, the β” precipitates coarsen and gradually transform into β’ precipitates (Figure 3 (c)) [12,13].

Figures 3 (d), (e) and (f) are micrographs of the underaged, peak-aged and overaged T6I6/177 microstructures, following the low temperature interrupt treatment. A high density of fine GP zones is observed following 6 hours of re-ageing at 177˚C. Very fine β” precipitates were observed in the peak aged microstructure after 15 hours of re-ageing. Further ageing caused coarsening of these precipitates and formation of β’ rods. These observations show that the T6I6/177 heat treatment produced visibly finer and more densely distributed precipitates than in the T6 peak-aged condition.

An even higher level of refinement in the microstructure was produced by the T6I6/150 ageing regime. A large number of very fine GP zones, shown in Figure 3 (g), were observed after 6 hours of re-ageing at 150˚C after the interrupt treatment. After 15 hours of re-ageing, a high concentration of fine precipitates, identified as GP zones and an early form of β” precipitates, were still present in the microstructure (Figure 3 (h)). A very high density of precipitates and the lower re-ageing temperature reduced the kinetics of transformation causing the very fine precipitates to dominate the microstructure even in a highly overaged T6I6/150 condition (Figure 3 (i)).

Further TEM observation was conducted in order to explain the origins of refinement in the microstructure of the T6I6 tempers. Figure 4 (b) reveals contrast arising from very fine GP zones formed during 20 minutes of initial ageing at 177˚C as compared with microstructure of the as-quenched sample shown in Figure 4 (a) where no precipitates were observed. Both hardness measurements and TEM observations indicate that a considerable amount of solute remained in solid solution after the initial ageing at 177˚C which underwent secondary precipitation over the 2 weeks of interrupted ageing at 65˚C.

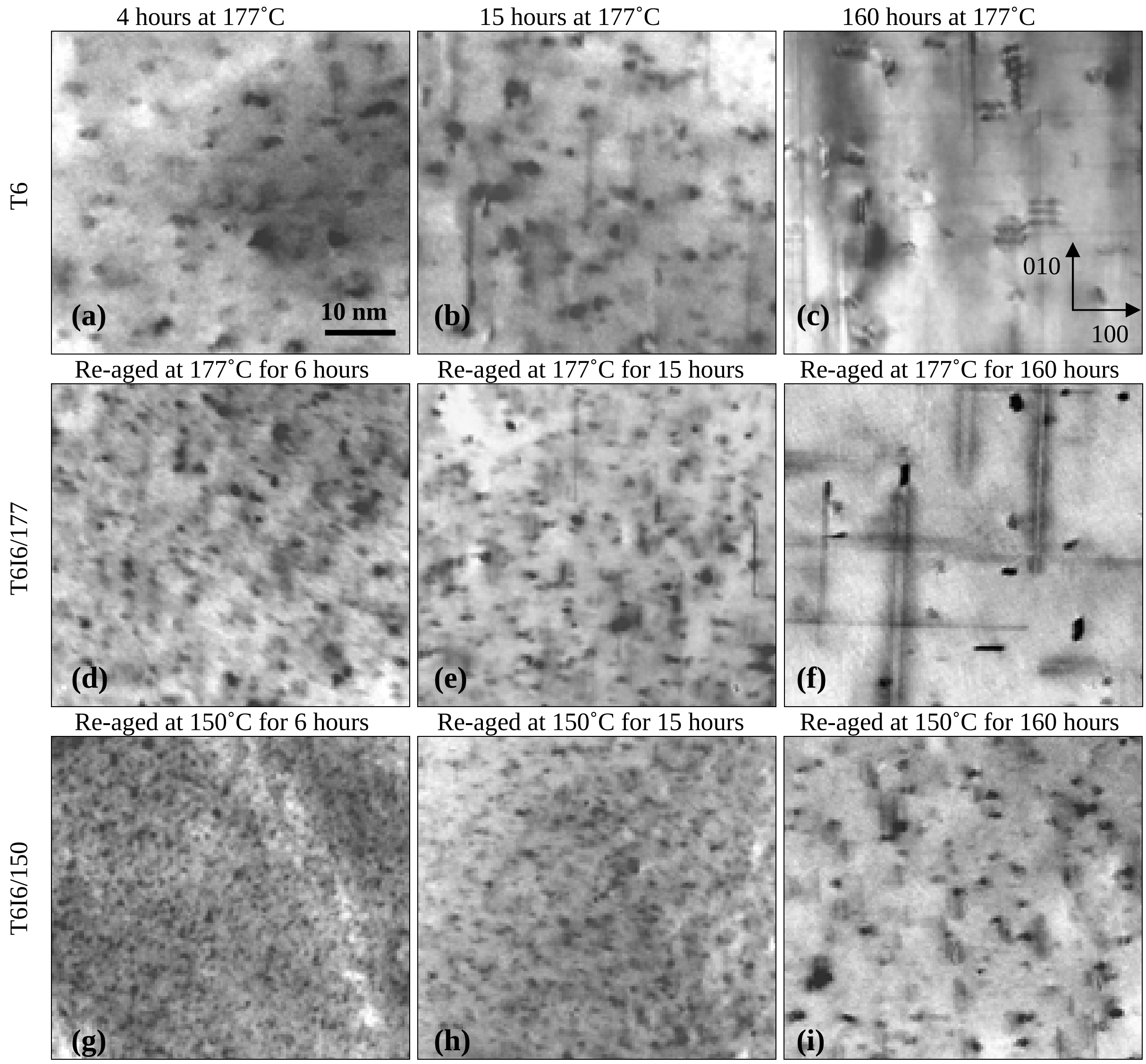

Figure 3: Bright-field TEM micrographs of 6061 taken with [001] matrix orientation from (a), (b) and (c) T6 tempers aged for 4, 15 and 160 hours respectively; (d), (e) and (f) T6I6/177 tempers re-aged at 177˚C for 6, 15 and 160 hours respectively following the initial ageing and interrupted ageing stages of the heat treatment; (g), (h) and (i) T6I6/150 tempers re-aged at 150˚C for 6, 15 and 160 hours respectively following the initial ageing and interrupted ageing stages. All micrographs were taken at the same magnification.

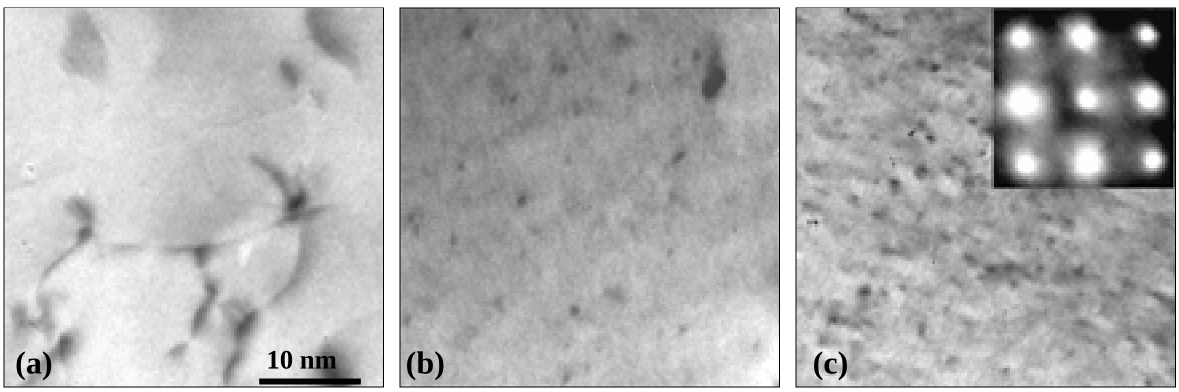

Figure 4: The development of microstructure during initial ageing and interrupted ageing: a) as solution treated and quenched, b) after 20 min ageing at 177˚C and c) after 20min at 177˚C and 2 weeks at 65˚C and a section from the corresponding SADP.

An increase in hardness of 12 Vickers was recorded over the 2 weeks period. Figure 4 (c) shows a high concentration of very fine precipitates in microstructure after the 65°C dwell period. The corresponding selected area diffraction pattern (SADP) did not show any evidence of the formation of β” precipitates. This implies that the precipitates formed during secondary ageing are GP zones.

It appears that GP zones formed through the process of secondary precipitation at 65˚C also attain a sufficient stability over the 2 weeks period such that they do not dissolve during the subsequent re-ageing stage of the T6I6 heat treatments. Moreover, these GP zones formed during interrupted ageing at 65°C act as precursors for nucleating a high density of the β” precipitates on re-ageing at 150 or 177°C.

4. Conclusions

The novel T6I6 heat treatment in which elevated temperature ageing is interrupted by a dwell period at 65°C causes an overall improvement in the mechanical properties of the commercial alloy 6061 when compared with those resulting from a conventional T6 temper.

1. Insertion of interrupted ageing into the conventional ageing treatment of 6061 caused a notable improvement in the tensile properties (T6I6/177 ageing cycle increased the 0.2% proof stress by ~ 8%) and also increased hardness.
2. The two variants of the T6I6 ageing treatments produce improvements of 21% and 36% to the damage tolerance of the alloy.
3. The improvements in mechanical properties are associated with refinement of the microstructure. This change is achieved through secondary precipitation of a very high density of GP zones during the dwell period at 65°C which then serve as precursors for the formation of a fine dispersion of the main strengthening phase, β”, when elevated temperature ageing is resumed.

Acknowledgements

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