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Impact of pre-ageing on age hardening response of twin-belt cast AlMg1SiCu sheet

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Abstract The potential of twin-belt cast (TBC) AlMg1SiCu sheet for structural automotive applications was investigated with a particular emphasis on the impact of pre-ageing on its age hardening response. The optimum T6 process for the TBC AlMg1SiCu sheet is identified to be a water-quench from the solution heat treatment at 540 °C and a subsequent ageing treatment at 180 °C. This process gives hardness values as high as 120 HV within several hours when ageing at 180 °C is performed shortly after the solution treatment. The age hardening capacity is impaired, however, when the sheet is stored at room temperature before the artificial ageing cycle. Pre-ageing at 100 and 140 °C is effective in improving the age hardening response of the naturally aged 6061 sheet. Pre-ageing suppresses natural ageing and clustering activities and gives lower T4 yet a much higher T6 hardness.

Introduction

Growing public awareness on environmental issues and the legislation addressing fuel consumption and exhaust emissions both mandate weight reduction in passenger cars [1–3]. Major weight savings is possible only through the use of light materials in structural body applications where high strength, good formability, weldability and corrosion resistance are primary requirements [1, 4]. Aluminium currently offers the greatest potential in this respect owing to its ability to reduce vehicle's weight without a loss in performance [5]. With only one-third the density of steel and a better strength-to-density ratio, aluminium could provide weight savings of about 50% when used in place of steel in automotive sheet applications.

While AlMg and AlMgSi sheet are technically very attractive, they suffer from high cost which is three to four times that of steel, their major competitor. This high cost has been the major impediment to the widespread use of aluminium sheet in high-volume automotive applications [6]. The high cost of aluminium alloys can be circumvented by cutting processing and/or material costs. Production of aluminium sheet by the strip casting rather than the conventional DC casting and hot mill route offers to reduce processing costs [7]. The twin-roll casting process has recently been shown to be a very attractive processing route for the manufacture of AlMg and AlMgSi sheet for automotive structural applications [8–19]. The twin-roll cast (TRC) AlMg and AlMgSi alloys were shown to be at least equivalent to, and in some cases superior than, their DC-cast counterparts [8].

Material cost is linked with the limitation of impurities which is quite low in the popular AlMgSi (6016 and 6022) alloys. That of Fe is particularly important since the ductility deteriorates due to coarse Fe-bearing intermetallics when the Fe content is above 0.2 wt%. The AlMg1SiCu alloy, which offers very good corrosion resistance, weldability and adequate cold formability in the T4 temper, is much more tolerant to Fe (Fe content is allowed to be as much as 0.7 wt%) [20]. Ductility impaired due to coarse intermetallic particles can be largely recovered by employing high solidification rates during casting. Owing to cooling rates, over an order of magnitude higher than those encountered in the conventional DC casting route, the strip casting process is attractive not only from processing but also material cost aspects. The present work was undertaken to explore the potential of the twin-belt cast

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(TBC) AlMg1SiCu sheet for structural automotive applications with a particular emphasis on the impact of pre-ageing on the age hardening response of TBC AlMg1SiCu sheet.

Experimental

AlMg1SiCu alloy (Table 1) was cast industrially with a twin-belt caster in the form of 600 mm wide, 18 mm thick strips and subsequently hot rolled to 10 mm. They were cold rolled to produce 1 mm thick sheet in the laboratory. Processing for the T6 temper involved a 2-h solution heat treatment at 540 °C before artificial ageing. Two quench practices, in water and in air, and three artificial ageing temperatures, 160, 180 and 200 °C were employed for T6 processing. The age hardening performance of the sheet thus obtained was investigated both in the freshly solutionized and in the naturally aged conditions by measuring the Vickers hardness of samples immediately after quenching and after holding at room temperature for 7 days and subsequently ageing at 180 °C.

Pre-ageing treatments at 100 and 140 °C for 10 min were employed to investigate their effect on the age hardening response. Samples processed with and without pre-ageing were held at room temperature for 7 days before they were finally submitted to artificial ageing treatment.

Microhardness, electrical conductivity measurements and differential scanning calorimetry (DSC) tests were employed to investigate the mechanisms responsible for natural ageing and age hardening and to identify the impact of pre-ageing on the age hardening response. A microhardness tester was used with 1000 g load and a dwell time of 15 s. A Sigma Test Unit measured the electrical conductivity of the samples to determine the clustering, precipitation and dissolution activities. DSC analyses were performed on 3 mm discs using a Setaram Labysys model DSC unit. A pure aluminium disc of equal mass was used as the reference. The cell was equilibrated first at 20 °C and then heated to 600 °C at 10 K min^{-1} in a dynamic argon atmosphere (1 L h^{-1}). The heat effects associated with transformation reactions were then obtained by subtracting a blank run from a given heat flow curve.

Table 1 Chemical composition of the EN AW 6061 alloy used in the present work

Si	Fe	Cu	Mn	Mg	Cr	Ti	Al
0.698	0.532	0.238	0.013	1.025	0.048	0.022	97.41

Results and discussion

The DSC scan of the fully solutionized AlMg1SiCu sheet samples (Fig. 1) reveals a series of exothermic signals which agree reasonably well with the well-established precipitation sequence for Al–Mg–Si alloys: supersaturated solid solution → vacancy-Si clusters → GP-I zones → β'' → β' → β [21–27]. The first exothermic peak which centres around 90 °C (signal 1) is linked with the formation of vacancy-Si clusters [24, 27]. The weak signal between 150 and 200 °C (signal 2) is associated with the formation of GP-1 zones. The much smaller size of signal 2 with respect to signal 1 implies that the majority of the GP-1 zones form through the evolution of vacancy-Si clusters. The next two neighbouring exothermic peaks between approximately 200 and 325 °C (signals 3 and 4) are produced by the precipitation of the β'' and β' phases, respectively, as suggested in previous studies on AA6061 monolithic and composite alloys [21, 23, 27]. The following relatively weaker exothermic signal centring around 350 °C (signal 5) is attributed to Si precipitation [28]. This latter peak is often missing in 6061 alloys [29] and is encountered only when there is sufficient amount of excess Si to precipitate. The last peak (signal 6) is associated with the precipitation of the equilibrium β-Mg_2Si phase.

The DSC spectrum of the sheet sample quenched in air following the solution treatment generally reveals similar features (Fig. 1). Several differences noted in the low temperature regime are accounted for by the partial decomposition of the supersaturated solid solution during the slower air-quench. The exothermic peak associated with the formation of clusters is missing suggesting that the supersaturation of the matrix is partially relaxed during cooling from the solution heat treatment in air. Air-quench cannot retain as much Mg and Si in solution as in

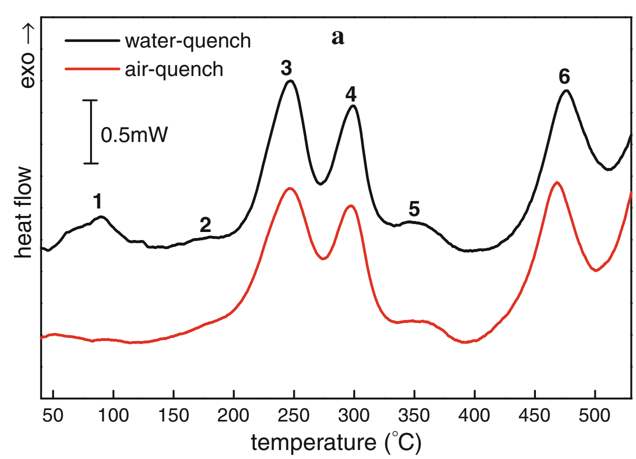

Fig. 1 DSC scan of the solutionized TBC AlMg1SiCu sheet samples quenched in water and in air upon artificial ageing

water-quench, thereby reducing the driving force for clustering activities at low temperatures. Signal 2 for the formation of GP-1 zones, on the other hand, has merged with signal 3 suggesting that the aluminium matrix of the air-quenched sheet contains stable zones which grow readily to become the coherent β'' particles during a subsequent ageing treatment.

While the features of the respective DSC scans are similar at typical artificial ageing temperatures, a clear distinction in the age hardening response was noted with respect to the quenching practice (Fig. 2a). Water-quench has invariably produced higher T6 hardness values for the entire range of ageing times. The age hardening capacity of the air-quenched sheet samples was relatively lower and could not be compensated by employing higher artificial ageing temperatures and longer ageing treatments. The electrical conductivity values of the water- and air-quenched samples are also distinctly different. Those of the latter are consistently higher contrasting the relatively lower hardness values (Fig. 2b). A water-quenched sample aged at 160 °C for 8 h, for instance, enjoys the highest T6 hardness value but has one of the lowest conductivity values measured (Fig. 2a, b). Well known to be an indirect measure of the solute content, these electrical conductivity measurements attest to the fact that the amount of precipitation is not necessarily proportional to the extent of precipitation hardening. This is further confirmed by the

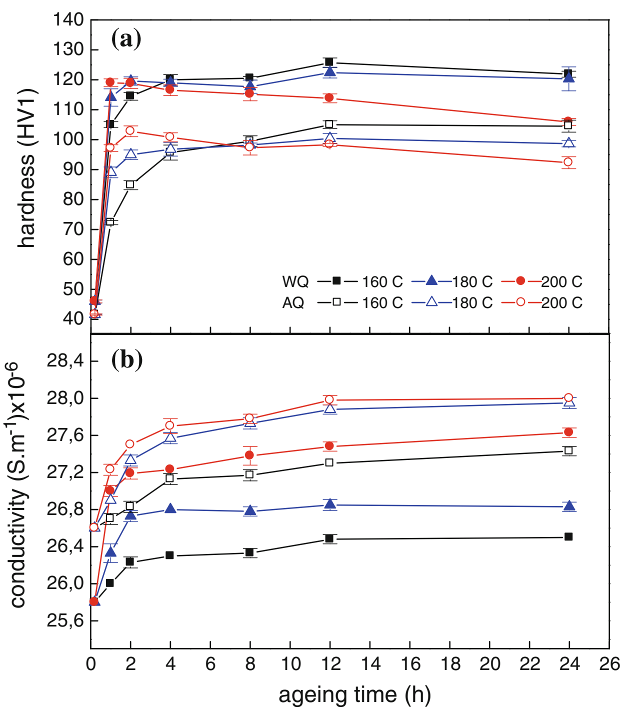

Fig. 2 Change in **a** hardness and **b** electrical conductivity of the solutionized TBC AlMg1SiCu sheet samples quenched in water and in air upon artificial ageing

dark-field optical micrographs which show extensive precipitation in the relatively softer air-quenched sheet samples after ageing at 180 °C (Fig. 3). It is indeed the quality rather than the quantity of the precipitating species that ensures age hardening. The precipitates coarse enough to be resolved readily with an optical microscope are clearly not as effective as those which are not directly evident in Fig. 3.

Quenching from the solution treatment was judged from the foregoing to be a highly critical processing step in the manufacture of AlMg1SiCu sheet with a big impact on the T6 hardness. The optimum T6 treatment is identified to be a water-quench from the solution heat treatment at 540 °C and a subsequent ageing at 180 °C. The hardness of the AlMg1SiCu sheet, measured to be 50 HV shortly after solutionizing, increased to 120 HV within an hour, when aged at this temperature immediately after the solution heat treatment (Fig. 2a). However, ageing to similar strength levels was not possible when the artificial ageing treatment was performed after room temperature storage. The hardness of the solutionized sheet has increased by approximately 50% to 75 HV after 7 days at room temperature, but was merely 80 HV, much less than the optimum T6 hardness, when subsequently aged at 180 °C for 1 h (Fig. 4a). The negative impact of natural ageing on the age hardening capacity in Al–Mg–Si alloys is well documented [18, 30–32].

The hardening during natural ageing implies clustering. The electrical conductivity values which were found to decrease with time at room temperature is a further evidence of the clustering activities (Fig. 4b) [33–35]. The supersaturation of the matrix was apparently relaxed completely by the formation of clusters and zones during natural ageing as inferred from the lack of the exothermic peak centring around 90 °C (signal 1) in the DSC curve of the freshly solutionized sample. These clusters and zones must first revert back into the matrix for the β'' phase to form. This reversion process is manifested in the DSC scan of the naturally aged sheet sample by a deep dissolution trough between 150 and 240 °C (Fig. 5). β'' precipitation is thus retarded as evidenced by the displacement of the β'' peak to higher temperatures. This is why the age hardening process is slowed down in the naturally aged sample thus giving a rather low hardness of about 80 HV after the 30 min at 180 °C.

Natural ageing was reduced, however, when 10-min pre-ageing treatments were employed at 100 and 140 °C before the sheet samples were stored at room temperature. Pre-aged samples have also experienced some hardening but were still softer after 7 days than those processed without pre-ageing (Fig. 4a). It is thus fair to conclude that pre-ageing at 100 and 140 °C suppresses subsequent clustering and associated hardening as confirmed by the electrical

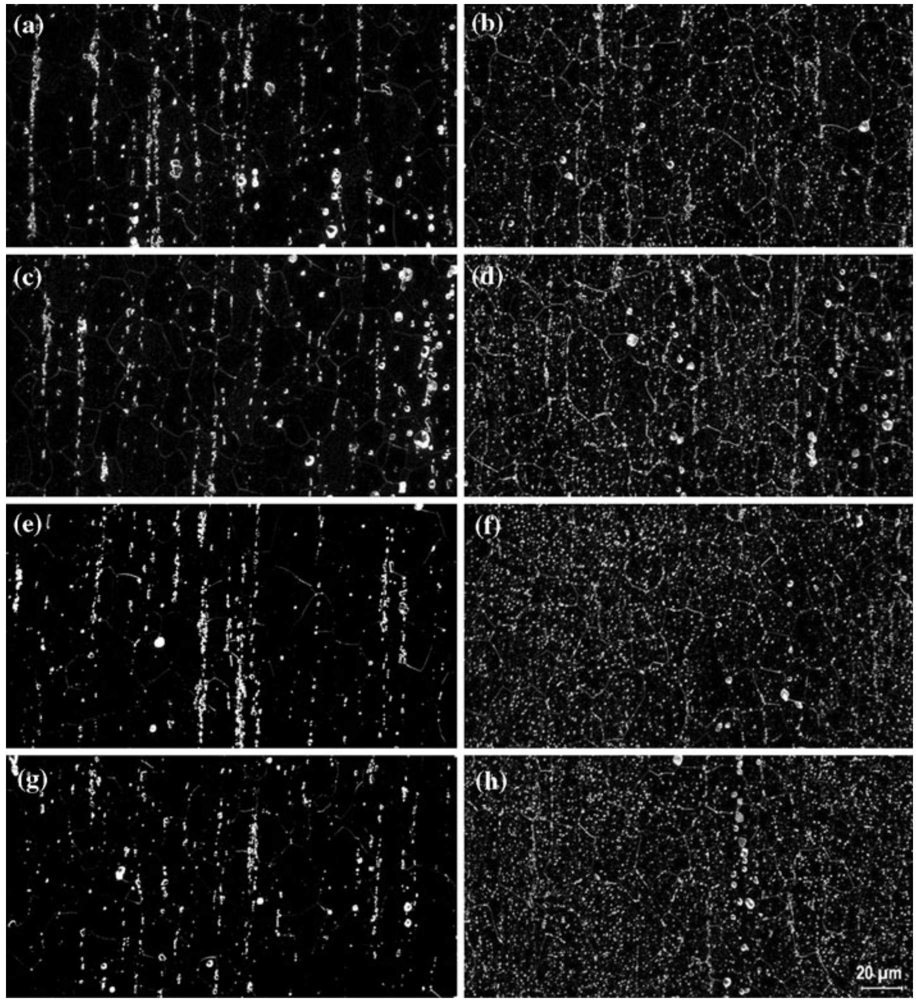

Fig. 3 Dark-field optical micrographs of the solutionized TBC AlMg1SiCu sheet samples quenched in water (**a**, **c**, **e**, **g**) and in air (**b**, **d**, **f**, **h**) and subsequently aged at 180 °C for 1 h (**a**, **b**), 2 h (**c**, **d**), 4 h (**e**, **f**) and 8 h (**g**, **h**)

conductivity measurements. The drop in electrical conductivity, a measure of the intensity of the clustering activities, was reduced in pre-aged samples with respect to the naturally aged sheet (Fig. 4b). The conductivity drop after pre-ageing at 140 °C was smaller than with pre-ageing at 100 °C, as one would expect. Clustering activities were apparently still in progress during natural ageing following pre-ageing in this temperature range but not as extensively as in natural ageing alone.

Clusters which form during natural ageing consume both quenched-in vacancies and solute atoms and are thus difficult to revert during artificial ageing, degrading the age hardening capacity of the alloy. Hence, suppressing natural ageing and decreasing the population of clusters and zones thus formed, not only provides a lower T4 hardness, but also improves the age hardening response of the sheet samples submitted to pre-ageing. The age hardening response improves with increasing capacity of the pre-ageing treatment to suppress the natural ageing process and is thus higher in sheet samples pre-aged at 140 °C.

The effect of low temperature pre-ageing on the bake hardening is demonstrated quite convincingly by DSC analysis (Fig. 5). The DSC scans of the sheet samples processed with and without pre-ageing are markedly different. The dissolution trough around 200 °C, ascribed to the reversion of the clusters and zones, is no longer evident in samples processed with a pre-ageing treatment at 140 °C suggesting that clusters are replaced by stable zones which readily transform into the hardening β'' precipitates during heating. β'' precipitation during subsequent ageing is thus accelerated in pre-aged samples. Pre-ageing at 140 °C apparently does more than just suppressing natural ageing. The β'' peak was observed to be reduced in size in the DSC curve of those samples pre-aged at 140 °C, implying that part of the precipitation activities responsible for this exothermic peak has taken place prior to DSC heating.

Conclusions

T6 hardness as high as 120 HV is readily achieved when water quenching is employed in the T6 process of the twin-belt cast AlMg1SiCu sheet. Sheet samples quenched in air, on the other hand, exhibit hardness values around 100 HV

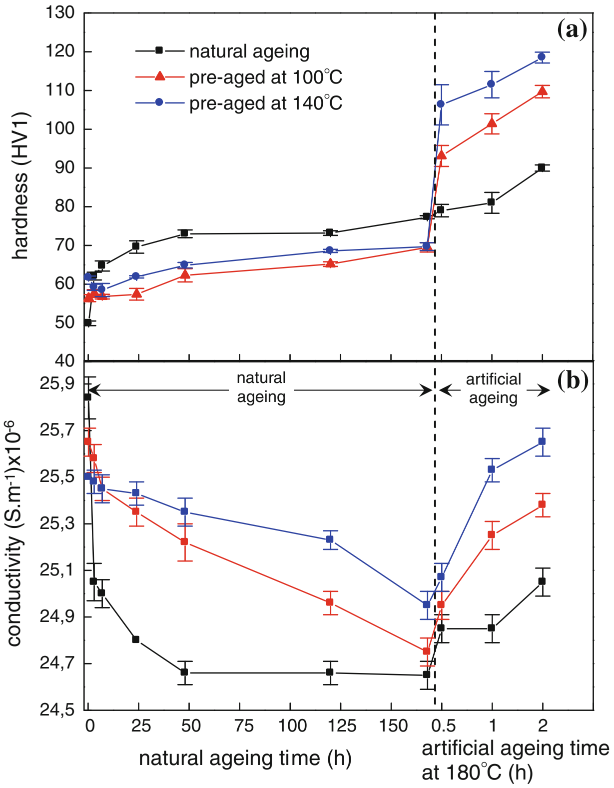

Fig. 4 Change in **a** hardness and **b** electrical conductivity of TBC AlMg1SiCu sheet samples processed with and without pre-ageing after solution heat treatment, held at room temperature for 7 days and subsequently aged at 180 °C

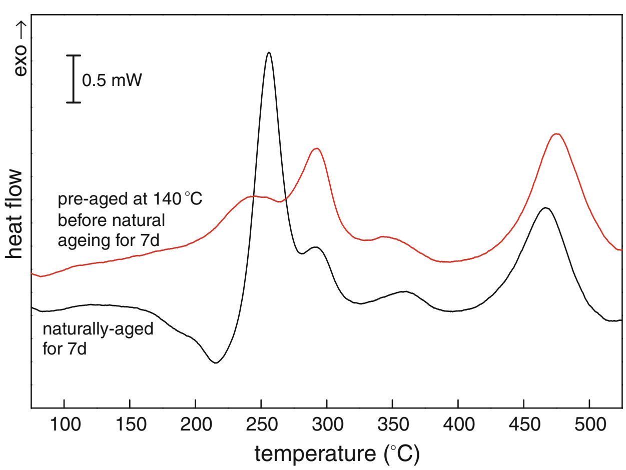

Fig. 5 DSC scans of TBC AlMg1SiCu sheet samples processed with and without pre-ageing after solution heat treatment, held at room temperature for 7 days

when submitted to similar T6 process. The optimum T6 process for the TBC AlMg1SiCu sheet thus involves a water-quench from the solution heat treatment at 540 °C and a subsequent ageing cycle at 180 °C. The age hardening capacity is impaired when the sheet is stored at room temperature before the artificial ageing cycle. Pre-ageing at 100C and 140 °C is effective in improving the age hardening response of the naturally aged TBC 6061 sheet. Pre-ageing suppresses natural ageing and the clustering activities and gives lower T4 yet a much higher T6 hardness.

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