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Double aging and thermomechanical heat treatment of AA7075 aluminum alloy extrusions

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Abstract The effects of double aging and thermomechanical double aging on enhancing mechanical properties and accelerating the kinetics of precipitation in aluminum alloy AA7075 were studied by means of hardness tests, tensile tests, and transmission electron microscopy. Using the appropriate heat treatment schedule, the time to peak aging was reduced by a factor of up to 36 without substantial decrease in hardness or tensile properties. The use of appropriate double aging and thermomechanical double aging showed that there can be a significant impact on energy savings and productivity resulting from the accelerated kinetics of precipitation.

Introduction

Age-hardenable aluminum alloys can be hardened or strengthened by natural or artificial aging. Supersaturated solid solution (SS) is formed after the solution heat treatment and, upon aging at temperatures well below the solutionizing temperature, GP-zones and metastable coherent precipitates form to harden the alloy up to the peak condition, while stable and incoherent precipitates start forming in the overaged condition. The type of precipitation varies with alloy composition, e.g., in Al–Zn–Mg alloys, of which AA7075 is an example, although the precipitation sequence is complex [1] the generally accepted sequence is [2]

$$\text{SS} \rightarrow \text{GP-zones} \rightarrow \eta'(\text{MgZn}) \rightarrow \eta(\text{MgZn}_2)$$

The GP-zones and η' are thermodynamically metastable but they have a lower free energy than the preceding microstructure, i.e., the supersaturated solid solution. This kind of precipitation is possible since kinetically it is easier to overcome the small activation energy barriers for coherent precipitates rather than the large barrier for the equilibrium incoherent precipitate.

Other research suggests that there are two kinds of GP-zones, type I and II [3]. Type I zones form over a wide range of temperatures up to 150 °C independent of the quenching temperature, and type II zones form at temperatures above 70 °C and have a strong dependence on the quenched-in vacancy concentration. The precipitation sequence is then [3]:

$$\text{SS} \rightarrow \text{Vacancy-rich clusters (GP(I)-zones)} \rightarrow \text{GP(II)-zones} \rightarrow \eta'(\text{MgZn}) \rightarrow \eta(\text{MgZn}_2)$$

Although artificial aging is the most widely used method of improving the mechanical properties, a variety of processing methods were developed by different researchers to achieve this end [4–7]. One method, two-step aging treatment, involves solutionizing and quenching the alloy, then heating the alloy to a temperature lower than the solutionizing temperature and pre-aging for a given time, and finally aging at either the same or at a different temperature from the first aging treatment; this process is also called double aging (DA). Early studies [8] of DA treatment suggest that strain induced either by quenching (thermal strain) or by cold or warm working (mechanical strain) can significantly affect the precipitation phenomenon.

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Thermal strain is induced by quenching the alloy from either the solutionizing temperature or from the first aging temperature. Mechanical strain can be introduced into the alloy right after solutionizing and quenching or between the two aging treatments; this process can also be termed thermo-mechanical double aging (TMDA). According to many researchers [8–10] both DA and TMDA processes can improve hardness and tensile properties and these processes are employed commercially on a wide variety of nonferrous materials, including age-hardenable aluminum alloys, with varying degrees of success. The TMDA process produces a stable and uniform dislocation and precipitate structure which resists localized cyclic strain thereby improving the fatigue resistance [11]. Correct TMDA treatment results in strength similar to T6 condition and better stress corrosion properties than achieved by over aging as in the T73 condition [12].

Double aging in alloys with a hierarchy of metastable phases should be done by first aging at a low temperature relative to the second aging treatment, because once a more stable phase is formed (e.g., η') it is not thermodynamically possible to form GP-zones by aging again at a lower temperature [13]. Pre-aging an age-hardenable alloy at a lower temperature than the second aging temperature appears to raise the hardness of the heat-treated alloy appreciably more than a DA treatment at the same temperature. This might be due to the formation of either a dense GP-zone structure or very fine transition phase precipitates in the case of Al–Zn–Mg alloys which are beneficial for nucleation of η' precipitates during final artificial aging steps. The effect of initial aging on the second aging depends not only on the hardness but also on the stability of the hardening structure [8]. Using the same temperature for both aging steps would have a meager effect, as the only difference in the two steps is the amount of excess vacancies and the thermal strain due to the quench from the first aging step [8, 14]. Although an increase in hardness is achieved by DA, often the peak hardness achieved by DA is not reported in the literature when compared to the peak hardness of the low-temperature single age treatment.

Of the many different DA heat treatments studied, the process has not been optimized, especially in regard to practical considerations of time and temperature to achieve high productivity and energy savings in extrusion plants and rolling mills. Also, cold stretching 1–3% after solution treatment or cooling from an elevated temperature shaping process conducted before artificial aging is a common commercial practice, and the effects of this imparted cold work on DA need to be understood. This study was performed to understand the effects of aging time, aging temperature, and amount of deformation on the mechanical properties and kinetics of aging for DA heat treatment of AA7075.

Materials and experimental procedure

The alloy AA7075 was selected for this study. The nominal composition of the alloy is presented in Table 1. Specimens were cut from extruded bars of cross-sectional dimensions 5.08×0.635 cm^2. The specimens were solution heat treated and aged at different temperatures to determine the aging curves. All specimens were solution heat treated in air at 490 °C in a box furnace for 30 min and quenched in water. Then the specimens were artificially aged in an air furnace and quenched in water. To understand the aging behavior, Vickers hardness tests were performed. Five readings were taken on each sample after quenching, and each datum point plotted on the aging curves is an average of five values.

For the DA treatment, the specimens were artificially aged in an air furnace at two different temperatures, 121 and 171 °C. The first aging step was done at 121 °C for a certain time ranging from 4 to 240 min, and the samples were then water quenched. The second aging step was then done at 171 °C to determine the full aging curve.

For the TMDA heat treatments, intermediate cold deformations of 5, 25, and 50% were introduced by rolling on a two-high rolling mill. The heat treatment profiles of DA and TMDA are shown in Figs. 1 and 2, respectively.

Transmission electron microscopy (TEM) analysis was performed using a JEOL 3110 electron microscope. Self-supporting thin film samples were prepared by twin jet electropolishing. A solution of 2% perchloric acid ($HClO_4$) in methanol was used as the electrolyte and the temperature was maintained at −30 °C. The current density of the polishing process varied depending on the sample heat treatment.

Tensile tests were conducted on 3.2-mm-thick flat dog-bone specimens. The specimens had a gage length of 25 mm and width of 6.3 mm. Three samples were tested for each datum point, and the average values are presented in the following section.

Table 1 Chemical composition limits of alloys studied

Alloy designation	Alloy composition (wt%)								
	Si	Fe	Cu	Mn	Mg	Zn	Cr	Ti	Al
AA7075	0.40 max	0.50 max	1.2–2.0	0.3 max	2.1–2.9	5.1–6.1	0.18–0.28	0.20 max	Bal.

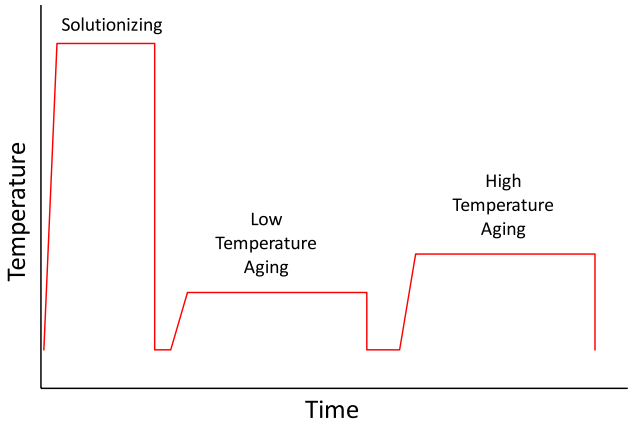

Fig. 1 Heat treatment profile of double aging (DA)

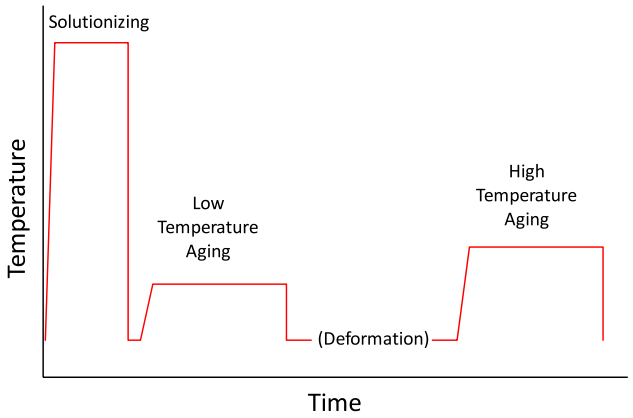

Fig. 2 Heat treatment profile of thermomechanical double aging (TMDA)

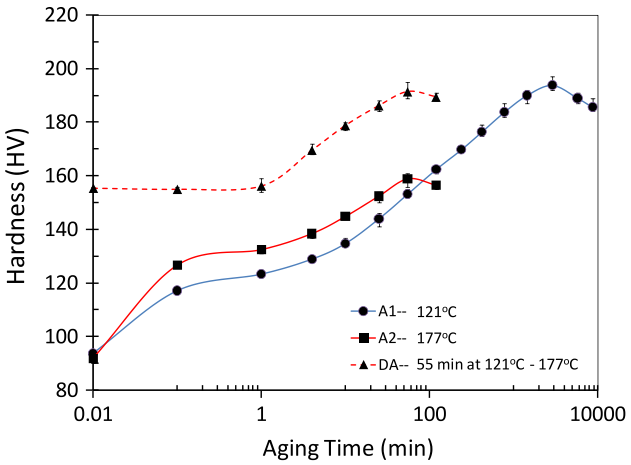

Fig. 3 Effect of single and double aging on hardness of AA7075 alloy. Note peak hardness of the DA is comparable to A1

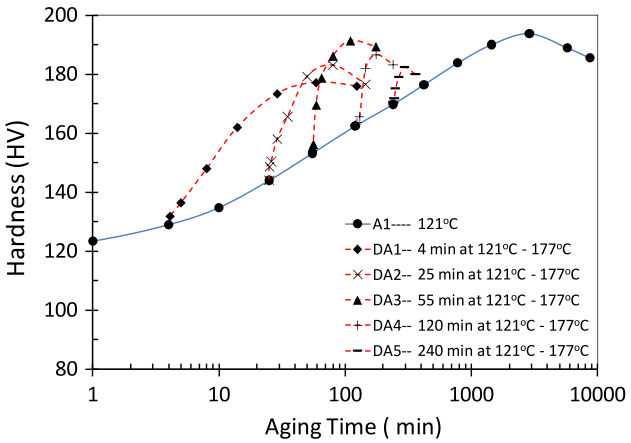

Fig. 4 Effect of time of first aging on double aging of AA7075 Alloy. Note the peak hardness of DA curves change with time

Experimental results and discussion

Double aging

For AA7075 alloy, the effect of heat treatment is significant, as the difference in peak hardness upon aging at 121 and 177 °C is large (Fig. 3) with the higher aging temperature producing significantly lower peak hardness. DA by initial aging at 121 °C for 55 min and subsequent aging at 177 °C results in a hardness comparable to the hardness obtained by single aging treatment at 121 °C (Fig. 3). A noteworthy observation is that the time taken to obtain the peak hardness is significantly reduced by the DA treatment, from 48 to 2 h (DA—1 h of initial aging and 1 h of final aging). Thus, the DA treatment can have a significant impact on energy savings and productivity in heat treatment of AA7075. Secondary aging at 177 °C leads to a rapid increase in hardness, indicating that this temperature is below the GP-zone solvus.

Perhaps the most important consideration for maximizing hardness in the DA treatment is controlling the time of the first aging treatment. Figures 4 and 5 show that by increasing the time in the first aging at 121 °C the peak hardness achieved after the final aging at 177 °C in the DA treatment increases at first but then decreases when the time of the first aging is longer than 55 min. Thus, aging for 4 min at 121 °C and then aging at 177 °C resulted in a peak hardness of 177 HV. Increasing the time of first aging to 55 min increased the final peak hardness to 191 HV; however, increasing the time of the first aging to 240 min decreased the peak hardness to 182 HV. The reason for this behavior is not yet determined but we can speculate that as the time of first aging increases, the volume fraction of precipitation increases, but after 55 min, the precipitate type changes, i.e., the GP zones transform to η' and coarsen. This affects the size and distribution of further precipitation at the second aging treatment.

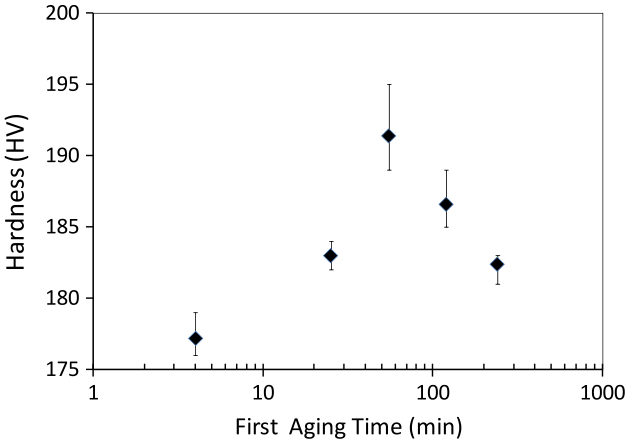

Fig. 5 Effect of time of first aging at 121 °C on peak hardness of second aging at 177 °C of AA7075 alloy. Note 55 min of first aging has the highest peak hardness after second aging

Thermomechanical double aging

Thermomechanical double aging led to an increase of hardness comparable to or higher than (depending on the amount of deformation) single aging at 121 °C. Larger amounts of cold work between the two aging treatments resulted in not only an increase in the initial hardness for the second aging treatment but also increased peak hardness as observed in Fig. 6. It can be seen in Fig. 6 that for 50% cold work the hardness was greater than that obtained by the DA treatment. This is due to a combination of strain hardening and dislocations acting as nucleation sites and resulting in a refinement of intermediate precipitates [10]. For smaller amounts of deformation, the peak hardness is lower than by DA treatment. Also, as seen in Fig. 6, another important aspect of TMDA treatment is that it has a pronounced effect on the acceleration of precipitation kinetics. The time to achieve the peak hardness for TMDA is reduced in the second aging treatment to 25 min rather than the 55 min it took for DA without the cold work.

For TMDA, the hardness of AA7075 upon aging does not increase for the first 10–20 min of second aging at 177 °C due to the recovery and precipitation mechanisms competing with each other, i.e., the recovery process reduces the hardness, whereas the aging process increases it [9]. After recovery, aging dominates, and the hardness increases. The peak hardness of TMDA is higher than for single aging treatment at 121 °C. This may be due to precipitation occurring on the dislocation cell boundaries that act as nucleation sites. Since a large number of dislocations are introduced throughout the matrix by the cold work, the precipitation in the dislocation sites could facilitate more finely dispersed precipitate structure for the same volume fraction [9]. However, as seen in Fig. 7, cold rolling just after solutionizing and before the first aging treatment (RA) neither increases the peak hardness beyond the hardness achieved by A2 (single aging at 177 °C) nor does it accelerate the kinetics of precipitation. Peak hardness of the curve designated RA (solutionized, rolled to reduce the thickness by 15%, aged at 177 °C) is similar to the A2 curve, which was aged in a single step at 177 °C. This behavior was also observed by other researchers in studies of aging phenomena in AA7075 alloy [9].

Microstructural analysis of double aging

The mechanism by which the DA treatment enhances the hardness and accelerates the kinetics of precipitation can be demonstrated by the results from the TEM. Figure 8 represents the microstructures of the single aging treatment at 121 °C for 48 h to peak hardness. We can observe from these microstructures that there is a very wide distribution

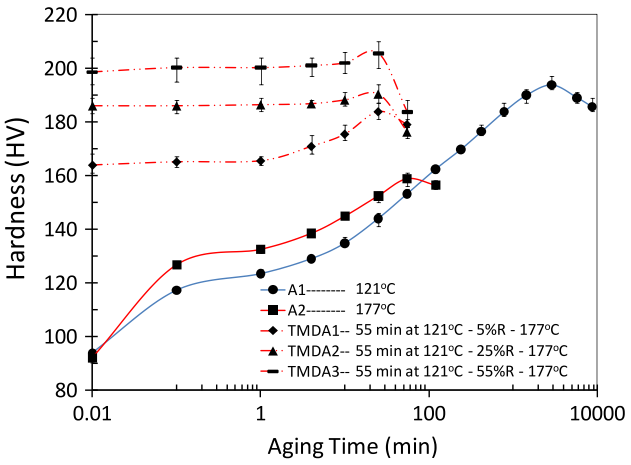

Fig. 6 Effect of TMDA on hardness of AA7075 alloy. Note the peak hardness of TMDA3 is higher than A1 and the line curve stays flat initially indicating simultaneous recovery and precipitation

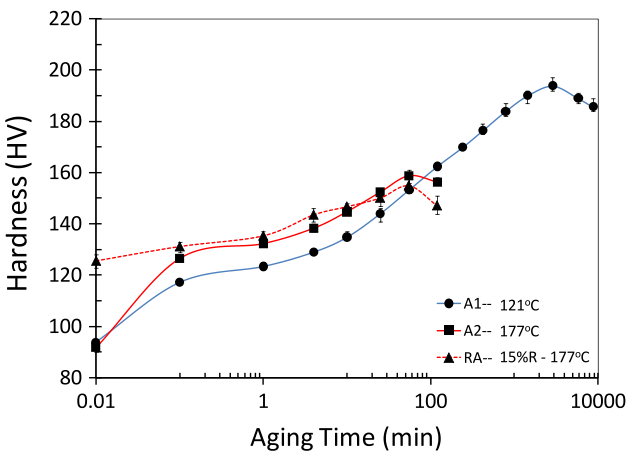

Fig. 7 Effect of rolling prior to single aging of AA7075 alloy. Note the decrease in peak hardness in RA

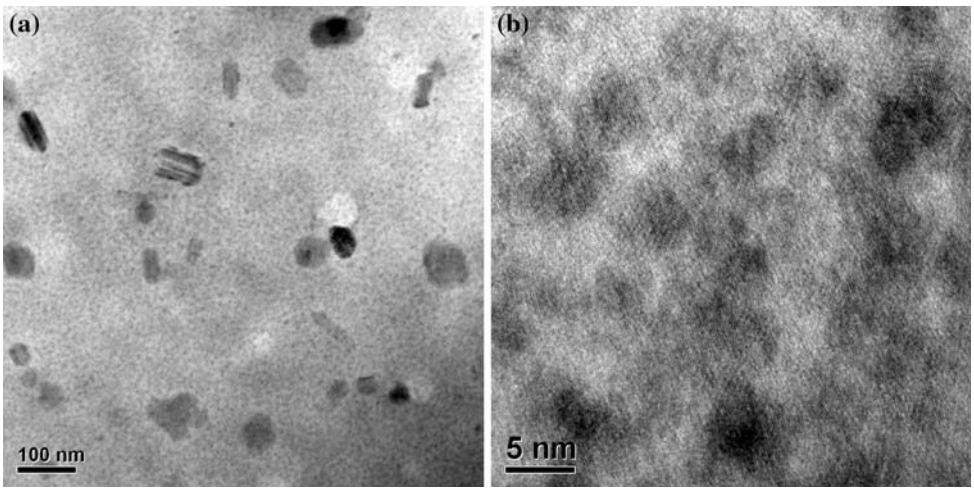

Fig. 8 TEM microstructure of single aging treatment at 121 °C for 48 h. **a** Image showing precipitates of fine transition phase or η' phase along with coarse E-phase dispersoids. **b** High-resolution image of fine η' precipitate or transition phase

of fine precipitates possibly η' or a transition phase from the GP-zones along with sparsely distributed coarse dispersoids called E-phase [15]. Our EDS analysis confirmed that the E-phase dispersoids are enriched in chromium and these have been shown not to dissolve during solutionizing [15]. The fine precipitates are generally accepted to be the primary cause for the hardness increase during the aging process.

In contrast to the 121 °C peak-aged structure, the single aging treatment at 177 °C for 55 min to peak hardness (Fig. 9) has a different microstructure of coarse hexagonal η' precipitates with a well-defined hexagonal disc

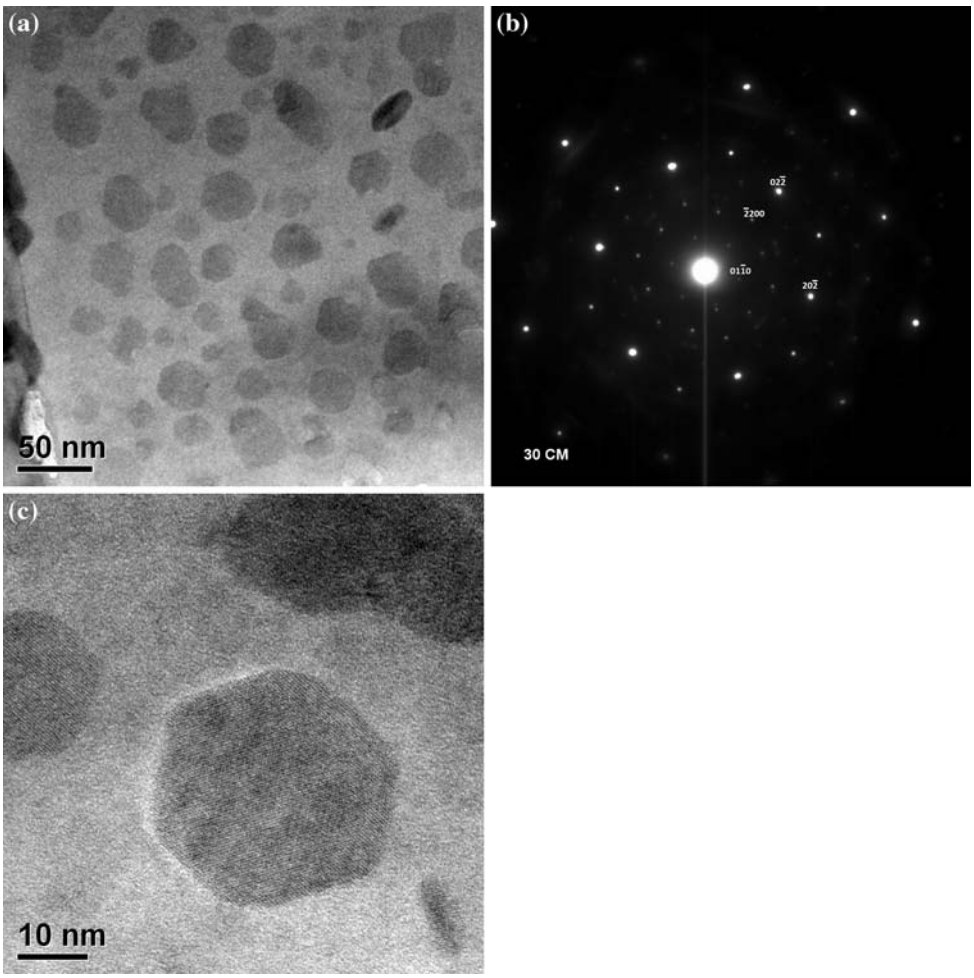

Fig. 9 TEM microstructure of single aging treatment at 177 °C for 55 min. **a** Image showing coarse hexagonal η' precipitates. **b** Diffraction pattern of the particles along with the matrix. **c** High-resolution image of η' precipitates

morphology. These precipitates have a $[111]_m//[0001]_p$ orientation relative to the matrix which has a lattice constant $a = 0.4078$ nm, while the precipitates in the peak-aged structure have a lattice constant $a = 0.5057$ nm (the lattice constant c was not determined). The precipitates formed upon aging at 177 °C are coarsened η' particles which have lost coherency. As expected, this microstructure has a lower hardness than the sample aged at 121 °C.

The microstructure for the DA treatment (55 min at 121 °C and 55 min at 177 °C) shown in Fig. 10 is very similar to the single aged heat treatment at 121 °C (Fig. 8), which explains the similarity in the peak hardness values. The size of the fine precipitate phase is approximately 5 nm in both heat treat conditions and the distribution of these precipitates appears identical. It has been suggested that these precipitates are a transition phase from the GP(II)-zones, enriched in zinc and internally ordered [3]. The additional spots observed on the diffraction pattern shown in Fig. 10c suggest that the precipitate structure does indeed have long range order. The super lattice spots do not appear in other orientations, suggesting an orientation relationship with the matrix such that solute enrichment occurs parallel to the {111} planes. The diffraction contrast would increase as the precipitates coarsen and completely transform from the transition phase to η' precipitates.

It has been suggested [16] that, in AA7075 alloy, the first aging in a DA treatment affects the radius of the GP zones and their volume fraction. Under the right conditions, the zones continue to grow initially during the second aging step, resulting in an in situ transformation to η' rather than the precipitation of a transition phase through nucleation on GP zones. Our TEM results show that the microstructure of both the single aging treatment at 121 °C for 48 h to peak hardness and the double aging (55 min at 121 °C and 55 min at 177 °C) is similar, which suggests that the first aging treatment forms GP zones and begins the transformation of these zones to η' and the second accelerates the conversion of the GP zones to η' precipitates. Detailed TEM analysis of this phenomenon will be reported in a subsequent paper.

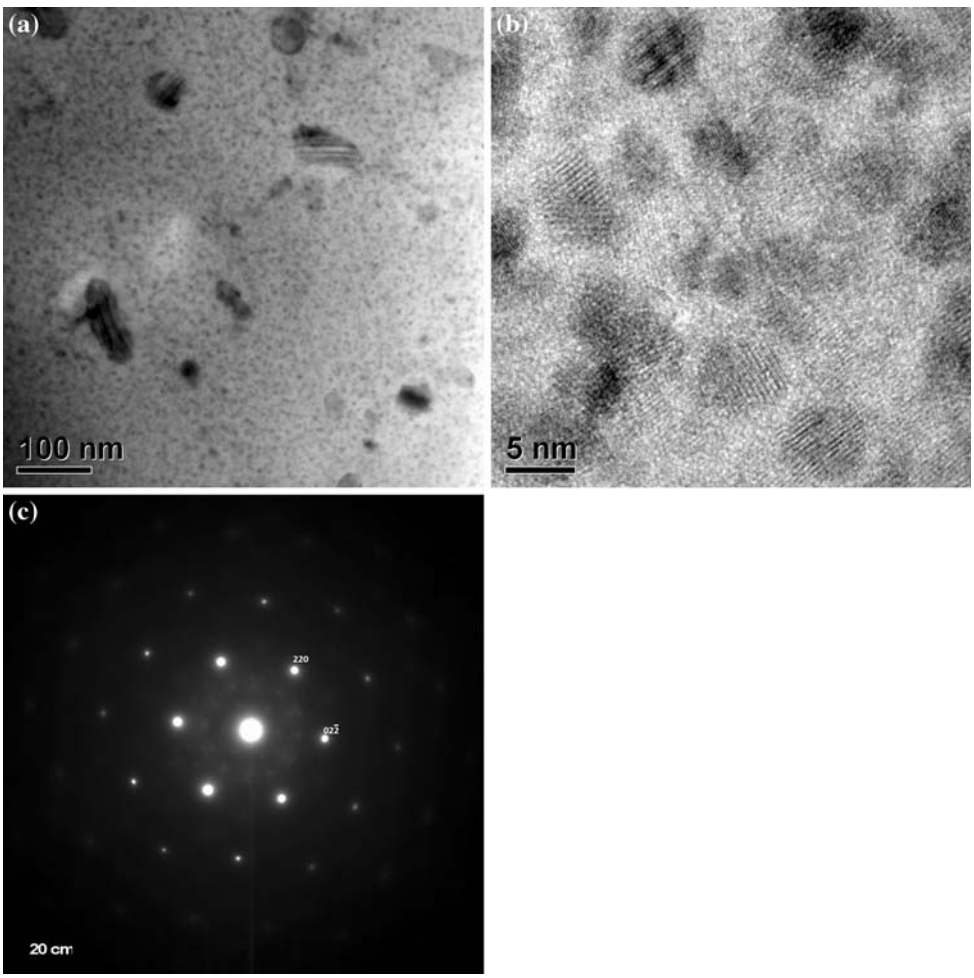

Fig. 10 TEM microstructure of DA treatment for 55 min at 121 °C and 55 min at 177 °C. **a** Image of the matrix showing the fine precipitates and a coarse E-phase along the grain boundary. **b** High-resolution image of fine η' precipitates or transition phase. **c** Diffraction pattern showing weak super lattice contrast from of the precipitates on {111} plane

Double aging process: effect of second aging temperature

The effect of temperature of the second aging treatment was also studied. The results show that, for the same conditions in the first aging treatment (121 °C, 55 min), the peak hardness for temperatures of 149 and 177 °C is similar, but the time to reach the peak hardness decreases with increase in temperature, as seen in Fig. 11. Final aging above a critical temperature for a constant first aging treatment results in loss of hardness, which is demonstrated in Fig. 12, where peak hardness achieved during the second aging at 204 °C is lower than that achieved during the second aging at either 149 or 177 °C. This is probably due to the dissolution of GP zones formed during the first aging step that are critical for peak age-hardening behavior. Thus, to achieve higher peak hardness in the DA treatment of AA7075, the second aging temperature should be selected below the GP zone solvus temperature.

Some work [17] suggests that the GP-zone solvus temperature is between 130 and 160 °C for Al–Mg–Zn alloys depending on the composition and as the Zn and Mg concentrations increase, the GP zone solvus temperature increases. For Zn and Mg contents of 5.5 and 1 wt%, respectively, the solvus temperature has been estimated as 160 °C. For the alloy under consideration, with 5.1–6.1 wt% Zn and 2.1–2.9 wt% Mg, the solvus is expected to be higher than 160 °C, and our aging results suggest it is between 177 and 204 °C.

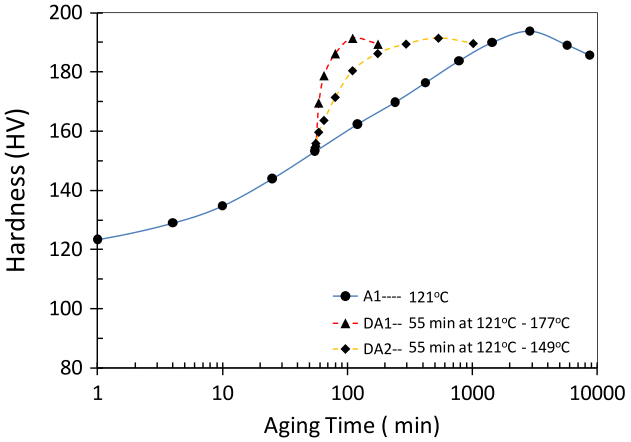

Fig. 11 Effect of second aging temperature on DA treatment of AA7075 alloy. Note the peak hardness of both DA treatments are the same

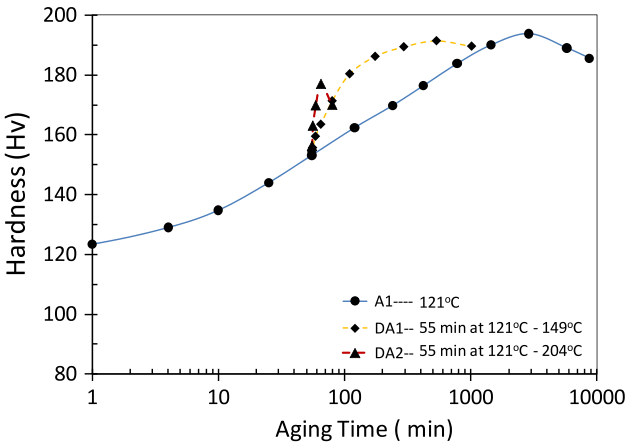

Fig. 12 Effect of second aging temperature on DA treatment of AA7075 alloy. Note the peak hardness of DA2 is lower due to the dissolution of the GP-zones

Mechanical property comparisons for different heat treatments

Representative engineering stress–strain curves for AA7075 alloy after A1, DA, and TMDA2 treatments are

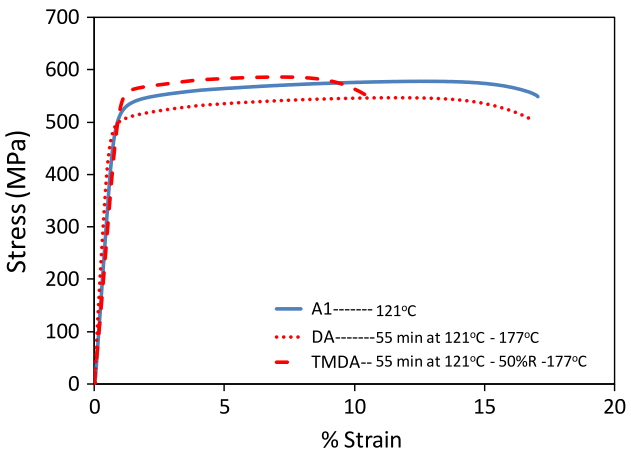

Fig. 13 Engineering stress–strain curves for AA7075 alloy after A1, DA, and TMDA2 treatments

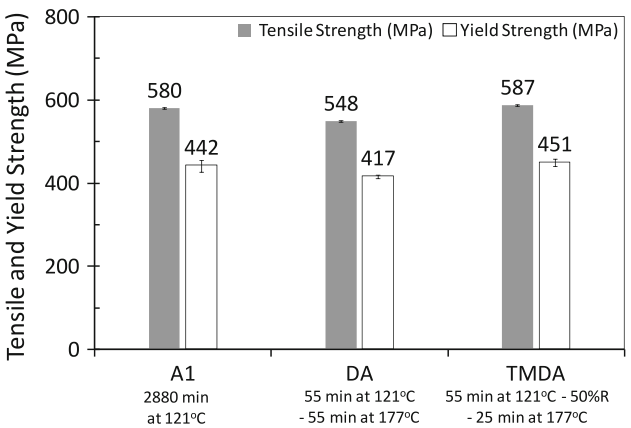

Fig. 14 Yield and tensile strength of AA7075 alloy after A1, DA, and TMDA2 treatments

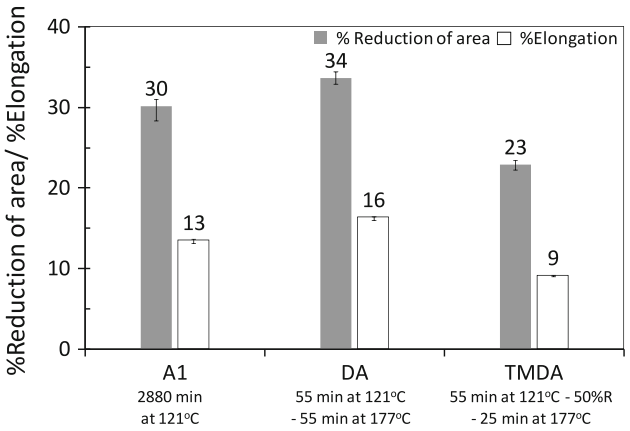

Fig. 15 Ductility of AA7075 alloy after A1, DA, and TMDA2 treatments

given in Fig. 13. The tensile strength and 0.2% offset yield strength of AA7075 in all conditions studied (single aging, DA treatment, and TMDA treatment) are shown in Fig. 14. Overall, the TMDA2 treatment resulted in higher strength than either A1 or DA treatments, which is consistent with the hardness results. The TMDA treatment had the lowest ductility compared with the A1 and DA treatments, which is due to the large amount of cold work introduced between the two aging treatments. The DA treatment resulted in the lowest strength but the highest ductility (Fig. 15). Nevertheless, we believe the modest decrease in strength for the optimum DA treatment is acceptable given the overall acceleration of the thermal processing.

Summary and conclusions

This study has established that DA can be used to accelerate the kinetics of precipitation in AA7075 alloy to achieve peak hardness. The proposed mechanism is that the first aging treatment forms GP-zones and begins the transformation of these zones to η', and the second accelerates the conversion of GP-zones to η' in AA7075 alloy, thereby reducing the time to achieve peak hardness. The DA accelerates the kinetics of precipitation by continuing the precipitation of η' from the GP-zones without changing the precipitate size, distribution, or morphology.

The most important factor influencing the DA treatment in AA7075 alloy is the time of first aging. There is an optimum time which may vary with the composition of the alloy and temperature of first aging treatment. A higher second aging temperature accelerates the precipitation in AA7075, but it cannot be so high as to cause significant reduction in volume fraction of precipitates or dissolution of GP-zones.

AA7075 showed positive effects of the DA treatment, with the ductility being higher for the DA treatment and a less than 6% decrease in yield and tensile strength. Double aging this alloy to peak hardness results in a significantly reduced processing time from 48 to 2 h and such processing can lead to reduced energy and cost in production.

Thermomechanical double aging causes further acceleration of precipitation requiring only a total heat treatment time of 80 min. This results in an increase in hardness and strength in AA7075 alloy beyond the conventional aging procedure but the ductility is reduced relative to conventional single age or the DA heat treatments.

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