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Improvement of mechanical properties of AA6063 aluminum alloy after equal channel angular pressing by applying a two-stage solution treatment

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Abstract

A two-stage solution treatment composed of soaking at 420 °C for 10 min and the second soaking at 500 °C for 10 min was applied to cold-worked AA6063 aluminum alloy samples after equal channel angular pressing (ECAP) for two and four passes. The microstructures and mechanical properties of the samples were compared with those of the samples after a routine one-stage solution heat treatment at 500 °C for 10 min. Abnormal grain growth (AGG) occurred to the samples during the one-stage solution treatment. However, no AGG was observed in the samples after the two-stage solution treatment. As a result of the prevention of AGG from occurring, the hardness, yield strength and ultimate tensile strength of the alloy after the two-stage solution treatment were significantly increased, while elongation to failure remained almost unchanged.

Keywords:

Aluminum; heat treatment; equal channel angular pressing; grain growth; mechanical property.

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1 Introduction

AA6063 is a heat-treatable medium-strength aluminum alloy with Mg and Si as the main alloying elements to contribute to its strength [1] through the formation of fine Mg_2Si precipitates during aging after a solution treatment. The routine solution treatment applied to this alloy prior to artificial aging is a single-stage treatment, i.e., soaking at a temperature above 500, e.g., at 520 °C, for 10 min or longer, depending on sample sizes, to ensure the dissolution of Mg and Si into the aluminum matrix [2]. It was observed in a previous study [3] that, during such a solution treatment at a temperature above 500 °C, abnormal grain growth (AGG) tended to occur to the alloy after severe plastic deformation, e.g., through equal channel angular pressing (ECAP), leading to the deterioration of mechanical properties such as strength, ductility, fatigue resistance and stress corrosion cracking resistance. It is therefore of great importance to find an effective way to avoid AGG by changing the routine solution treatment scheme.

In the case of hot-extruded AA6063 alloy, a separate solution treatment is often skipped by applying online quenching so that dynamically recrystallized grains have little possibility to grow. In this industrial practice, it is of critical importance to control the extrusion process in order to ensure the dissolution of maximum amounts of Mg and Si into the aluminum solid solution, when the hot-deformed product leaves the extrusion die and enters the online quench channel. The resultant mechanical properties after artificial ageing are affected by extrusion process parameters, such as billet temperature, extrusion speed, reduction ratio and extrusion die design [4–11]. Due to practical constrains, it is impossible to apply quench as soon as the extruded product leaves the die orifice and there is always a time interval between the moment that the extruded product leaves the die orifice and the start of an intensive cooling process. The interval time is usually sufficient to dissolve Mg_2Si particles into the aluminum matrix and yet short enough to avoid measureable grain growth. To avoid distortions of extruded products, air jet or mist is applied, although water quench delivers the best results in terms of microstructure and mechanical strength. In general, for hot-extruded AA6063 alloy subjected to online quench, AGG is rarely an issue.

In the case of cold-deformed AA6063 alloy, a separate solution treatment is usually applied to allow all Mg and Si to be dissolved into the aluminum matrix and in the meantime static recrystallization takes place. Depending on the extent of deformation and the scheme of the solution treatment, strong strain energy stored in the deformed material may drive recrystallized grains to grow abnormally, often locally, leading to the deterioration of mechanical properties. To achieve the best combination of strength and ductility, modification of the solution treatment from the routine scheme is necessary. Modified heat treatment schemes are generally considered proprietary and not discussed in the open literature.

In an effort to avoid the occurrence of AGG to cold-worked AA6063, a two stage solution treatment scheme composed of soaking at 420 °C for 10 min and the second soaking at 500 °C at 10 min was developed. It was found to be able to ensure the dissolution of sufficient amounts of the alloying elements into the aluminum matrix and in the meantime prevent AGG from occurring. The positive effects of such a treatment were evidenced by the mechanical properties of the samples both in the as-solutionized state and in the as-aged state.

2 Experimental procedure

The AA6063 aluminum alloy with a chemical composition shown in Table 1 was received in the form of hot-extruded rods with a diameter of 100 mm. Cylindrical samples with a diameter of 20 mm and a length of 120 mm were machined from the extruded rods to allow the samples to be inserted into the die of an equal angular channel pressing (ECAP) machine. Prior to ECAP, the samples were annealed at 550 °C for 30 min, leading to the formation of a coarse and fully recrystallized microstructure so as to eliminate any effect of previous deformation history on the metallurgical phenomena occurring during ECAP and subsequent heat treatment.

Table 1- Nominal and measured chemical compositions of the AA6063 alloy used in this investigation.

Element (wt. %)	Al	Mg	Si	Fe	Cu	Mn	Cr	Zn	Ti
AA6063 used in this study	Balance	0.84	0.57	0.31	0.03	0.04	0.03	0.10	0.03
Nominal composition of AA6063	Balance	0.45-0.9	0.2-0.6	Max 0.35	Max 0.1	Max 0.1	Max 0.1	Max 0.1	Max 0.1

An ECAP die with two channels of 20 mm in diameter intersecting at an angle of 90 degrees and outer curved corner of 22.5 degrees was used in this investigation. The samples were extruded up to four passes at a ram speed of 1 mm/s. After ECAP, the samples were solution-treated, using two different schemes. The first scheme was the conventional one-stage solution treatment, i.e., soaking at 500 °C for 10 min. The second scheme was a two-stage treatment, composed of soaking at 420 °C for 10 min and then the second soaking at 500 °C for another 10 min. After the solution treatments, the samples were quenched in water. Some of the samples were investigated in the as-solutionized condition and the rest were subjected to artificial aging at 175 °C for 8 h.

Microstructural investigation was performed on the longitudinal section of the samples. The grain structure was studied by using a polarized light microscope. The evolution of Mg_2Si particle distribution throughout annealing, ECAP, solutionizing and aging was revealed by optical microscopy and scanning electron microscopy (SEM). Both JEOL 6500 FEG-SEM and Tescan Vega SEM were used for this purpose.

Vickers hardness measurements were taken on the plane normal to the ECAP direction, using a load of 300 g. The measurements were repeated six times for each sample and the average value is presented in this paper.

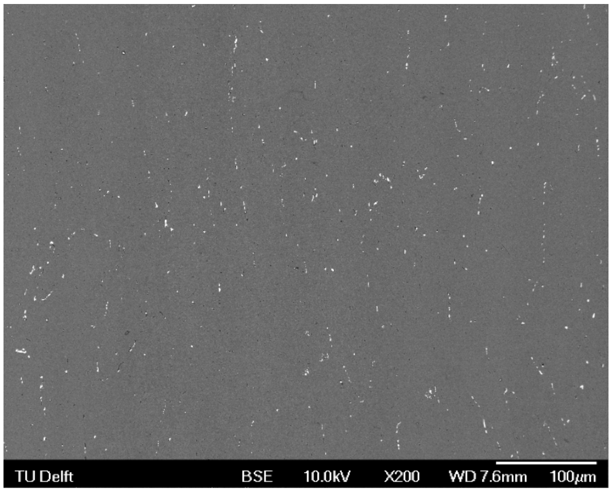
For tensile tests, sub-sized plate-like specimens were extracted from the samples, according to the ASTM E8 standard. All the dimensions were divided by two in order to facilitate the extraction of the tensile test specimens from the samples. The gauge length, width and thickness were 12.5, 3 and 2 mm, respectively. The total length of the specimen and the length of the grip section were 50 and 15 mm, respectively. Tensile tests were performed using a SANTAM tensile testing machine at a crosshead speed of 0.7 mm/min. The load-displacement data were registered. Engineering stress-strain data were calculated from the load-displacement values. Yield strength (YS), ultimate tensile strength (UTS) and elongation to failure (e_f) were determined. Note that YS was calculated using the 0.2% offset method according to the standard procedure.

3 Results and discussion

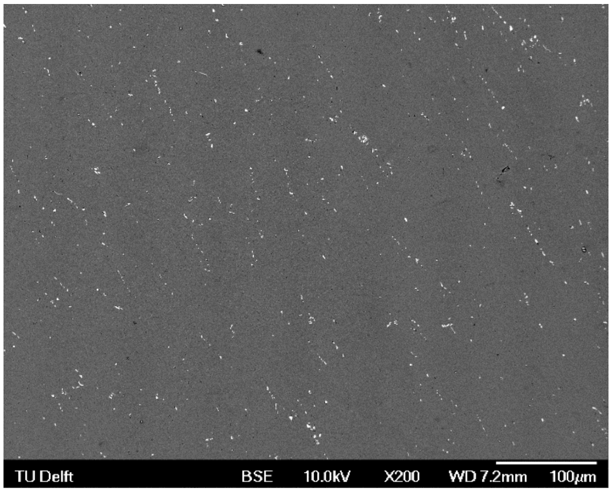
3.1 Microstructures before and after ECAP

3.1.1 Second phase particles

The main second phase in the AA6063 alloy is Mg_2Si particles [12]. After slow cooling in a furnace from the annealing temperature, these particles were coarse enough to be revealed by SEM. The distributions of Mg_2Si particles in the as-annealed state and after two and four passes of ECAP are shown in Figure 1. Image analysis indicated that the volume fraction of second-phase particles ranged between 4 and 5 %. This value as well as the distribution of Mg_2Si particles was not affected by ECAP up to four passes at room temperature, as visible by comparing Figure 1a, b and c. The average size of these particles was around 5 µm.


TU Delft
BSE
10.0kV
X200
WD 7.6mm
100µm

(a)


TU Delft
BSE
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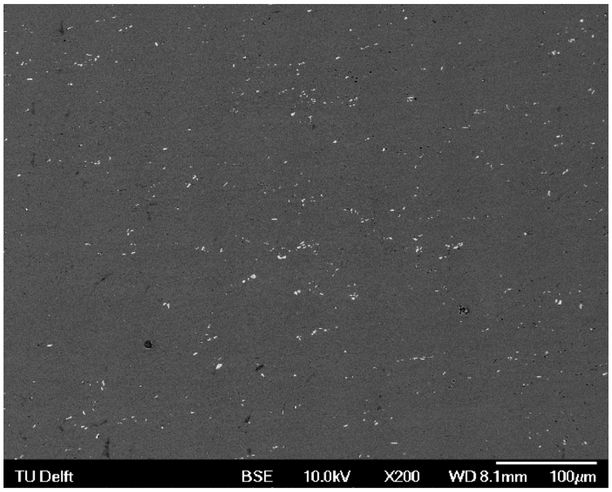
100µm

(b)


TU Delft
BSE
10.0kV
X200
WD 8.1mm
100µm

(c)

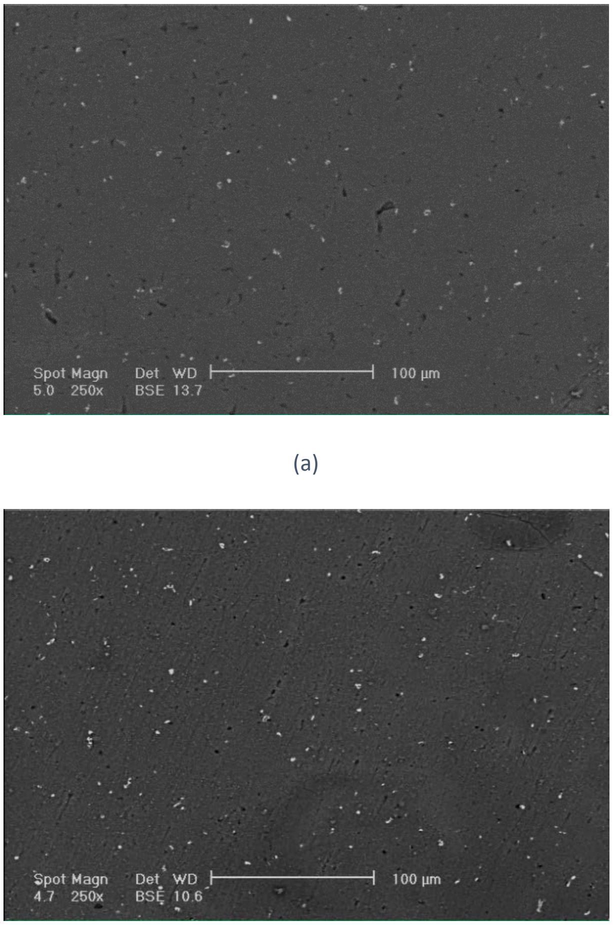
Figure 1 – Second-phase particles in (a) the as-annealed state and after (b) two passes and (c) four passes of ECAP.

3.2 Effect of the one-stage solution treatment

3.2.1 Second-phase particles

A one-stage solution treatment is routinely applied to the AA6063 alloy [2,13,14]. The aim of the solution treatment is to dissolve maximum amounts of the alloying elements into the aluminum solid solution prior to aging [12]. To achieve this aim, a maximum temperature just below the solidus temperature of the alloy, at which the solid solubility limits of the alloying elements are maximum, may be considered. However, a rise in the solution treatment temperature may lead to grain growth and even abnormal grain growth (AGG). It is therefore important to determine an optimum solution temperature, at which a considerable fraction of second-phase particles is dissolved, while a minimum extent of grain growth or no AGG occurs. The minimum temperature applicable for the solution treatment of this alloy is 500 °C, as used in this investigation. Obviously, in order to prevent AGG, the soaking time should be minimized as well. Previous investigations showed that AGG could be inhibited at 500 °C if the time of the treatment was reduced to 2 min [3]. This time was however too short for the dissolution of second-phase particles at this temperature. Thus, it appeared to be impossible to avoid AGG during the solution treatment of this alloy and in the meantime achieve full dissolution of second-phase particles. In the current investigation, as the samples were small, soaking at 500 °C for 10 min was applied first.

The effect of the one-stage solution treatment on the distribution of second-phase particles is shown in Figure 2. It can be see that in comparison with the microstructures shown in Figure 1, the volume fraction of second phase particles reduced. Image analysis showed that the volume fraction of second-phase particles reduced from about 5 % in the as-deformed condition to around 1 % in the as-solutionized condition. It was also observed that the remaining Mg_2Si particles became spheroidized with round edges. This suggested that the time and temperature of the solution treatment might be convenient to dissolve a reasonable fraction of Mg_2Si particles into the aluminum solid solution.



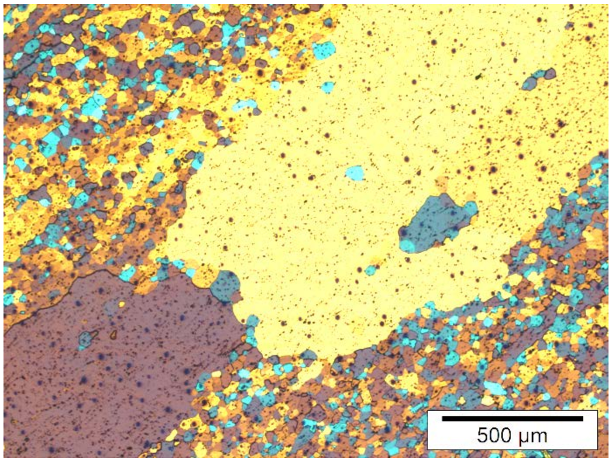
(a)

(b)

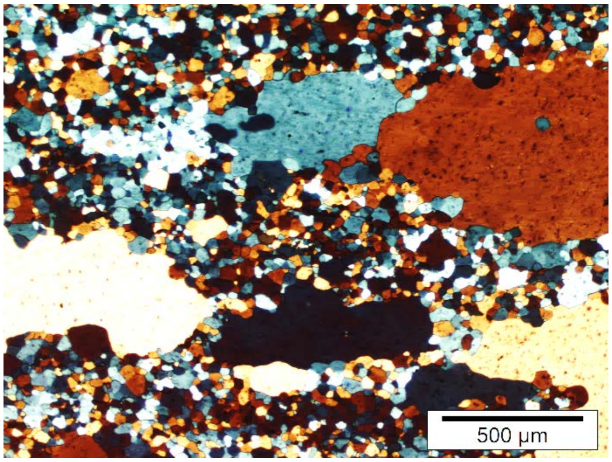
Figure 2- Effect of the one-stage solution treatment at 500 ^{0}C for 10 min on the distribution of second-phase particles after (a) two passes and (b) four passes of ECAP.

3.2.2 Grain structure

The effect of the one-stage solution treatment at 500 °C for 10 min on the grain structure of the samples after two and four passes ECAP is shown in Figure 3. It can be seen that none of the microstructures are lamellar and both are instead fully recrystallized. However, two different recrystallized regions are observed in each sample. A very fine region which is fully recrystallized without extensive grain growth and few extra-coarse slightly elongated grains which are attributed to the occurrence of abnormal grain growth (AGG). The difference between the two microstructures is that the one related to 2 passes ECAP shows more significant AGG than the one after 4 passes ECAP To understand how AGG occurred during the solution treatment, the mechanisms of recrystallization of a severely deformed aluminum alloy during high-temperature annealing may be considered. In fact, only a small number of nuclei can form during high-temperature annealing, because the new grains grow fast and consume the unrecrystallized area. These recrystallized grains grow fast inside a severely deformed structure due to a very high level of stored energy of deformation, the thermal activation energy provided by the high temperature and a lack of second-phase particles pinning the grain boundaries. In other words, if a few recrystallized grains are formed in the heavily deformed structure ahead to other recrystallized grains, there will be huge differences in grain size after the solution treatment.



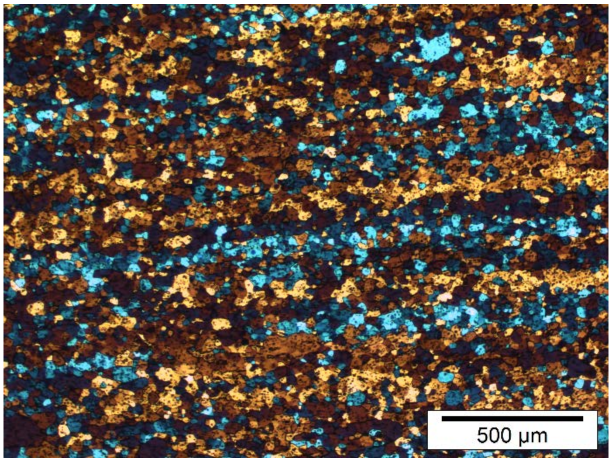
(a)



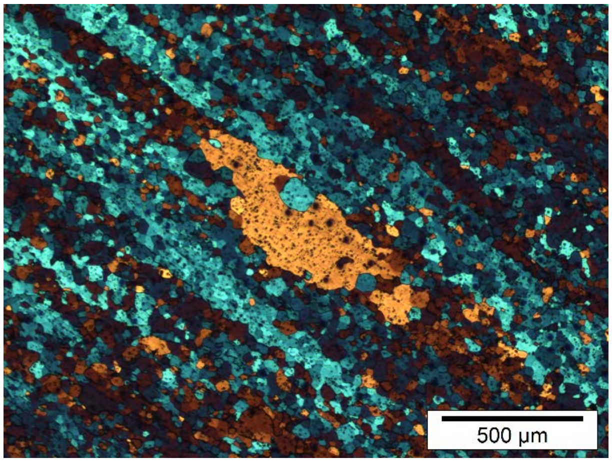
(b)

Figure 3- Microstructures of the samples after (a) two passes and (b) four passes of ECAP and then the one-stage solution treatment.

As the occurrence of AGG degrades the alloy both in strength and ductility, a reduction of soaking time would be the first measure to be taken. The effect of shorter soaking times, i.e., 2 and 5 min, on the microstructures of the samples after two passes of ECAP is shown in Figure 4. It was found that if the samples were soaked at 500 °C even for 5 min, AGG occurred. To avoid AGG, the solution treatment time must be reduced to 2 min. Similar results were obtained in a previous study on the samples subjected to four passes of ECAP [3].



(b)



(b)

Figure 4- Microstructures of the samples after two passes ECAP and one-stage solution treatment at 500 ^{0}C for (a) 2 and (b) 5 min.

To find out whether 2 min would be enough for the dissolution of second-phase particles, the distribution of Mg_2Si particles, as shown in Figure 5, was examined. It was found that the fraction of second-phase particles remained almost unchanged, as compared with that shown in Figure 1. Therefore, it was confirmed that with the one-stage solution treatment it would not be possible to achieve the dissolution of Mg_2Si particles and avoid AGG.

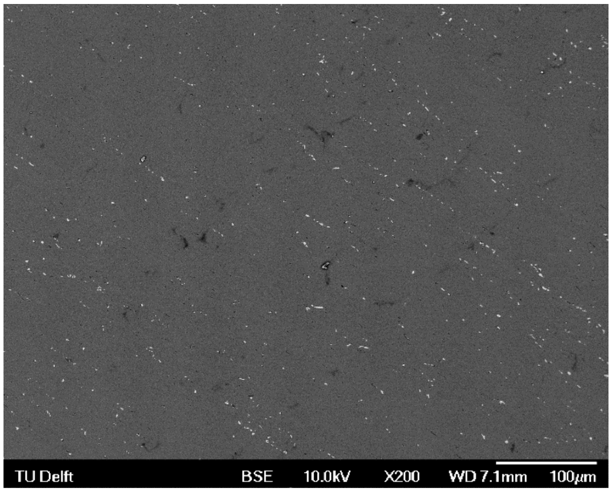


Figure 5- Effect of the one-stage solution treatment at 500 ^{0}C for 2 min on the distribution of second-phase particles after 2 passes of ECAP.

3.2.3 Hardness and tensile properties

The effect of the one-stage solution treatment on the hardness of the samples in the as-solutionized state and in the as-aged state is shown in Figure 6. It can be seen that the hardness values in the sample after four passes of ECAP, both in the as-solutionized state and in the as-aged state, were higher than the hardness values after two passes of ECAP. As all the samples were fully annealed prior to ECAP, no effect of prior deformation history on hardness was expected. The measurement of the average grain size indicated that the sample after four passes of ECAP had a finer grain structure, which corresponded to a higher hardness value, according to the Hall-Petch relationship [15]. (The average grain size was determined in the regions where no AGG was observed.) Obviously, higher hardness values of the as-aged samples, relative to those of the as-solutionized samples, could be attributed to the precipitation hardening effect by the formation of fine, coherent Mg_2Si precipitates [12,13].

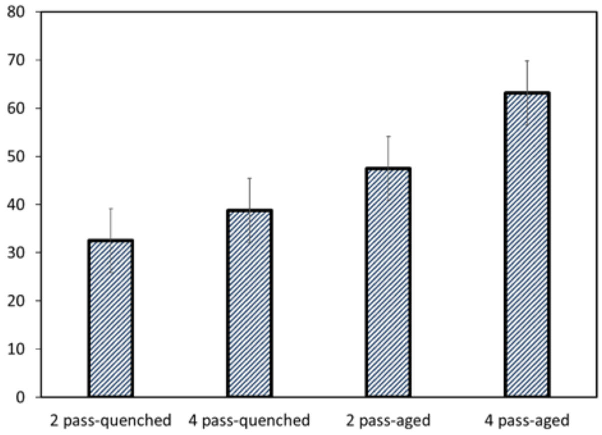
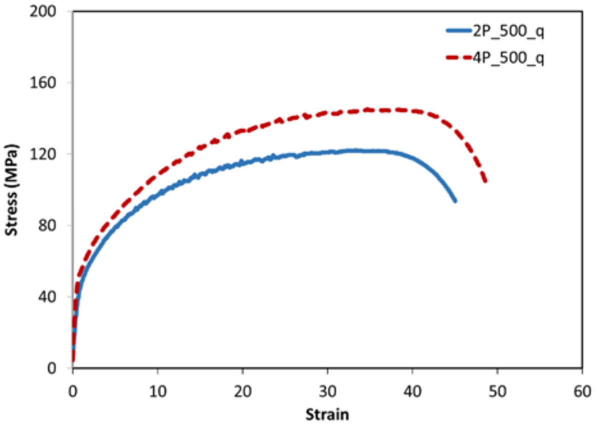


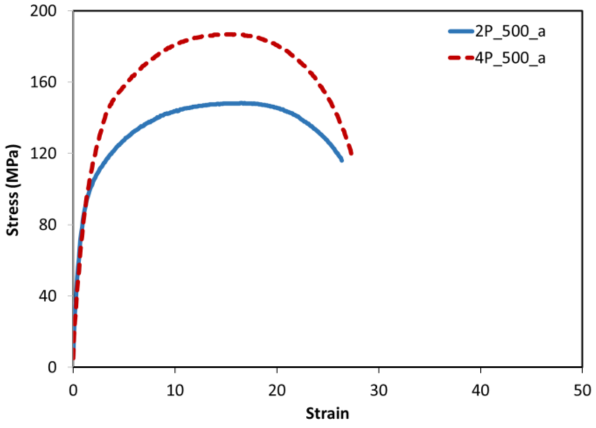
Figure 6- Effect of one-stage solution treatment on Vicker's hardness.

Engineering stress-strain curves are shown in Figure 7. A typical stress-strain curve of a stress-relived material with a recrystallized grain structure [15] was observed in Figure 7a. As observed in Figure 3, although AGG was found in all the samples, the grain structure was in general finer for the sample after four passes of ECAP. Therefore, a higher flow stress of the sample after four passes of ECAP than that of the sample after two passes of ECAP could be attributed to a finer grain structure of the former.

Similar observations were made of the samples in the aged state, as shown in Figure 7b. Indeed, the flow stress of the sample after four passes of ECAP was higher than that after two passes of ECAP. In addition, the flow stresses of the as-aged samples were in general higher than those at the as-solutionized conditions, which clearly indicated the effectiveness of the artificial aging applied. In addition, the elongation was significantly reduced, which could be attributed to more server work hardening occurring in the samples with nano-sized coherent precipitates formed during aging [15].



(a)



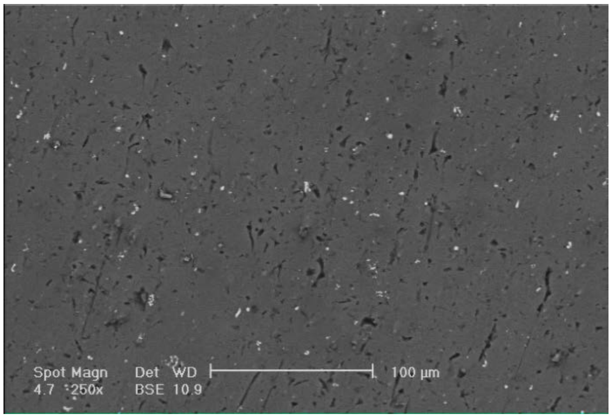
(b)

Figure 7- Engineering strain-stress curves of the samples in (a) the as-solutionized state and (b) the as-aged state after the one stage solution treatment.

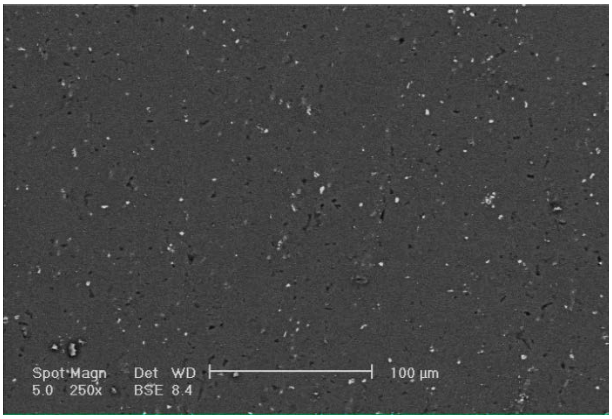
3.3 Effect of the two-stage solution treatment

3.3.1 Second phase particles

To avoid the negative effects of the one-stage solution treatment on the microstructure and mechanical properties of cold-worked AA6063, a two-stage solution treatment composed of soaking at 420 °C for 10 min and the second soaking at 500 °C for 10 min was developed. The effect of the two-stage solution treatment on the distribution of second-phase particles is shown in Figure 8. The fraction of Mg_2Si particles was measured to be around 1 %, being similar to the samples after the one-stage solution treatment. In fact, adding the first soaking at 420 °C caused no effect on the dissolution of Mg_2Si particles into the aluminum solid solution. This is because the dissolution of Mg_2Si particles is a thermodynamics- and kinetics-related process [16], both of which are strongly temperature dependent. Moreover, the solid solubility limits of the alloying elements increase with temperature from 420 to 500 °C. Therefore, more dissolution takes place at 500 °C than 420 °C. It should be noted that a previous investigation showed the precipitation of particles at 420 °C instead of dissolution [3]. Therefore, the second soak at 500 °C largely determines the dissolution of Mg_2Si particles.



(a) 1%+1.3%

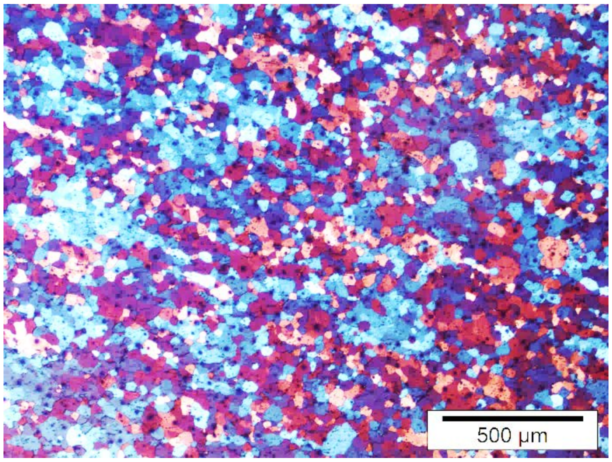


(b) 1.2+0.7

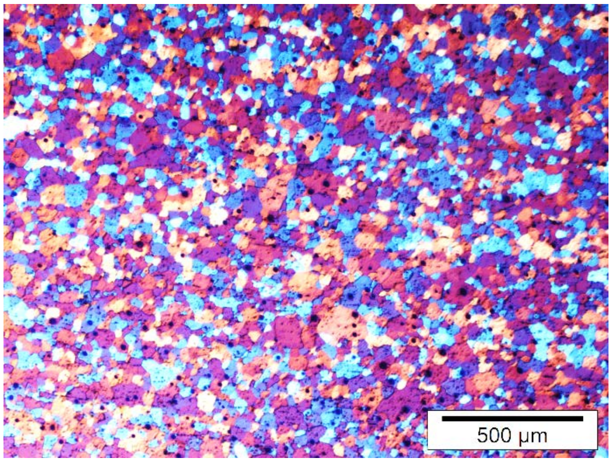
Figure 8- Effect of the two-stage solution treatment at 420 °C for 10 min, followed the second soaking at 500 °C for 10 min on the distribution of second-phase particles after (a) two passes and (b) four passes of ECAP.

3.3.2 Grain structure

The effect of the two-stage solution treatment on the grain structures of the samples after two and four passes of ECAP is shown in Figure 9. It can be seen that although the two-stage solution treatment had no effect on the distribution of Mg_2Si particles, it was highly effective in limiting grain growth. In fact, no AGG could be observed in the samples after the two-stage solution treatment.



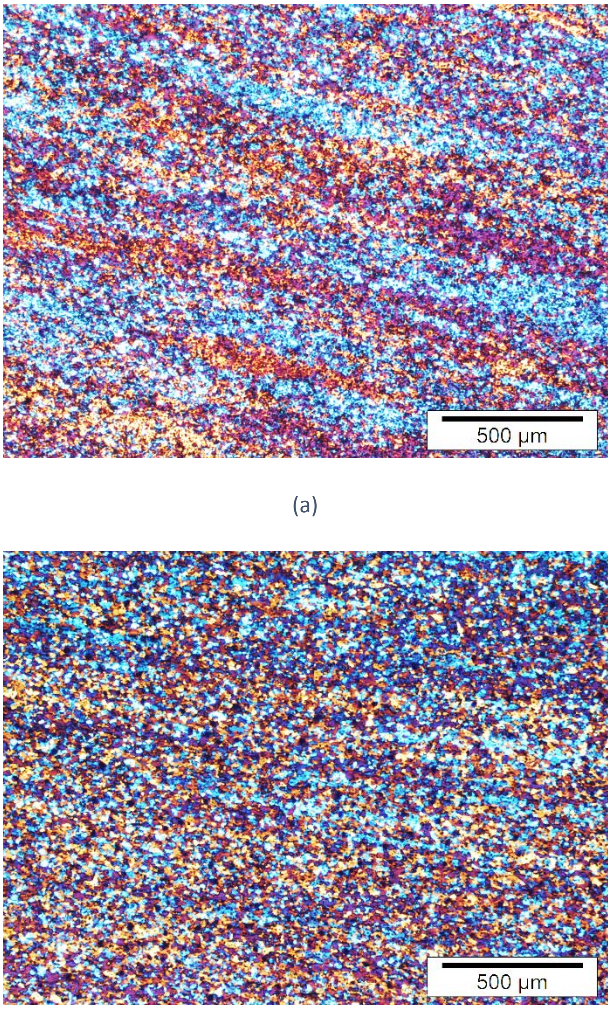
(a)



(b)

Figure 9- Microstructures of the samples after (a) two passes and (b) four passes of ECAP, followed by the two-stage solution treatment.

In order to understand the metallurgical phenomena occurring during the second-stage solution treatment, leading to the inhibition of AGG, the microstructures of the samples after the first soaking at 420 °C for 10 min were examined and these are shown in in Figure 10. As can be seen, both the samples were fully recrystallized and fine, uniform grain structures were formed. At this temperature, a much larger number of nuclei could emerge in the deformed structure, which resulted in a fine, uniform grain structure. By performing the first soaking at 420 °C, the stored energy of deformation got largely consumed. As a result, when the sample was heated further to 500 °C and soaked there for another 10 min, there was no stored energy any more for the abnormal growth of some of grains in the largely recrystallized matrix. In fact, only normal grain growth occurred, as visible from the comparison in microstructure between Figure 9 and Figure 10.



(a)

(b)

Figure 10- Microstructures of the samples after (a) two passes and (b) four passes of ECAP and the first soaking at 420 °C for 10 min.

3.3.3 Hardness and tensile properties

The effect of the two-stage solution treatment on the hardness of the samples in the as-solutionized state and in the as-aged state is shown in Figure 11. Similar results, as observed after the one-stage solution treatment, shown in Figure 6, were obtained. In fact, the hardness values of the samples in the as-aged condition and those of the sample after four passes of ECAP were higher. These phenomena could be attributed to precipitation hardening and finer grain structure of the samples after four passes of ECAP, respectively.

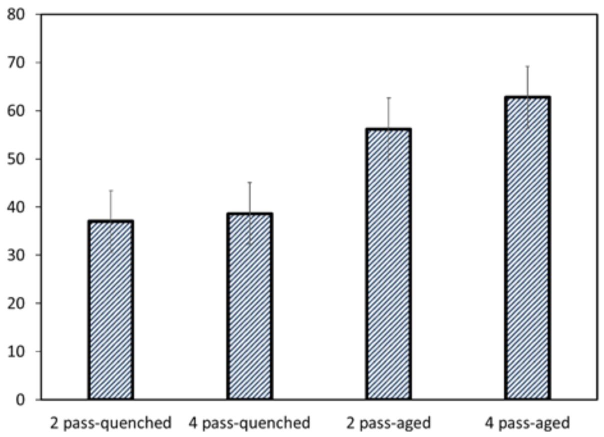
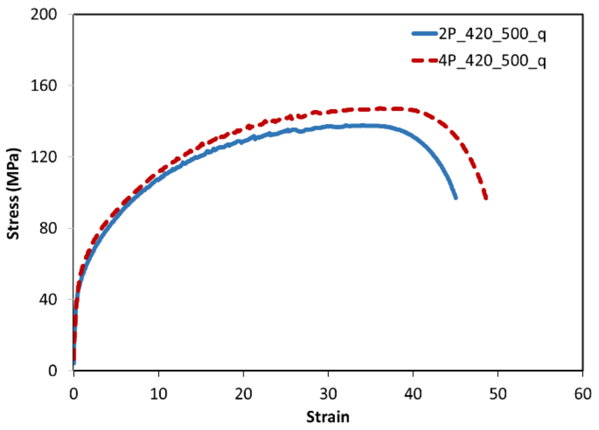
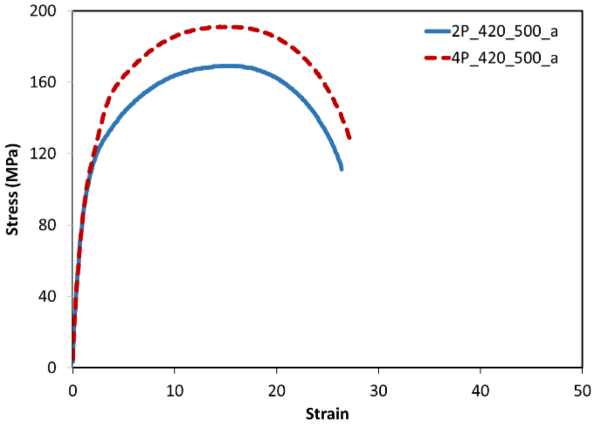


Figure 11- Effect of the two-stage solution treatment on the Vicker's hardness of the samples in the as-solutionized state and in the as0aged state.

The engineering stress-strain curves are shown in Figure 12. It is clear that in the as-solutionized state the yield and tensile strengths as well as the elongation of the samples after four passes of ECAP were higher than those after two passes of ECAP. Obviously, it was due to a finer grain structure of the samples after four passes of ECAP. Similar results were obtained for the samples in the as-aged state, as shown in Figure 12b.



(a)

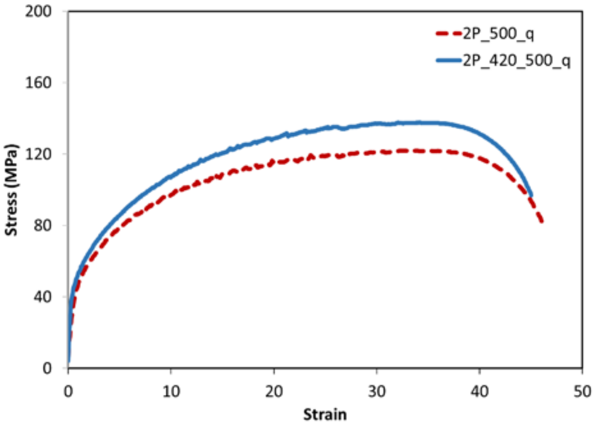


(b)

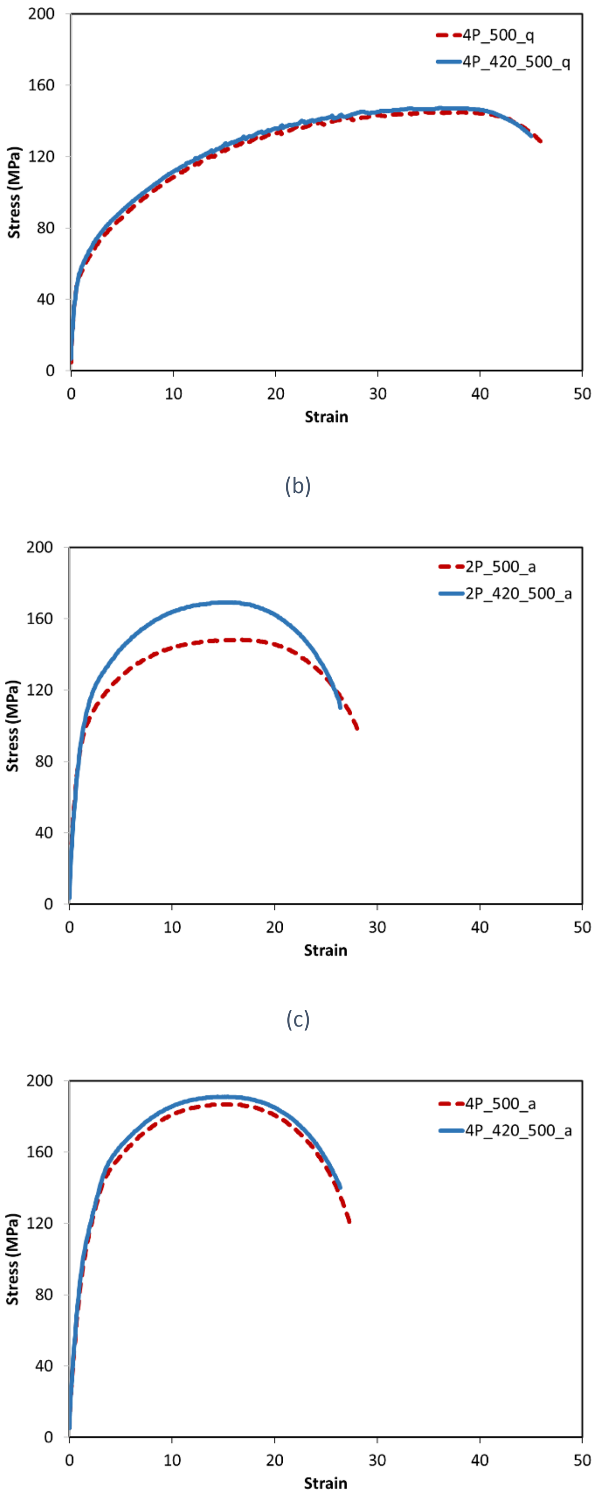
Figure 12- Engineering strain-stress curves of the alloy (a) in the as-solutionized state and (b) after two-stage solution treatment and aging.

3.4 Comparison between the one-stage and the two-stage solution treatments

The comparisons in the engineering stress-strain curves of the samples in the as-solutionized state and in the as-aged state between the one-stage solution treatment and the two-stage solution treatment are given in Figure 13. It can be seen that, in general, in the as-solutionized and as-aged conditions, the samples after the two-stage solution treatment were stronger. However, the difference was negligibly small for the samples after four passes of ECAP, while the difference was larger for the samples after two passes of ECAP.



(a)



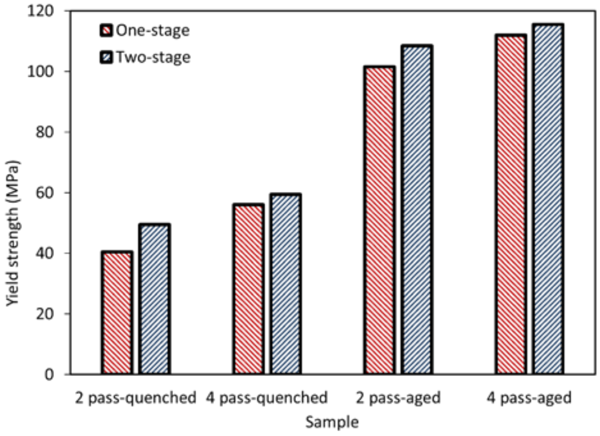
(b)

(c)

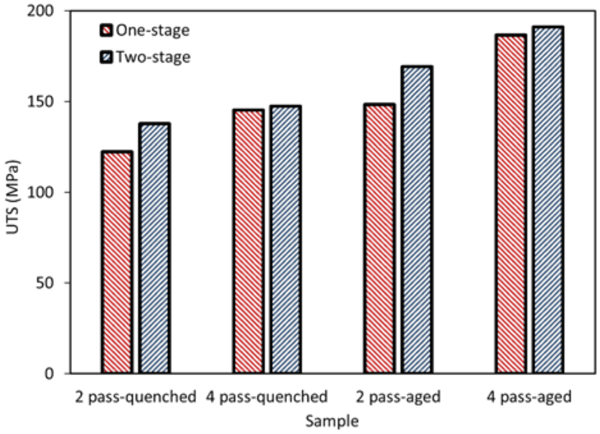
(d)

Figure 13- Effects of the one- and two-stage solution treatments on the tensile properties of the samples (a) in the as-solutionized state after two passes of ECAP, (b) in the as-solutionized state after four passes of ECAP, (c) in the as-aged state after two passes of ECAP and (d) in the as-aged state after four passes of ECAP.

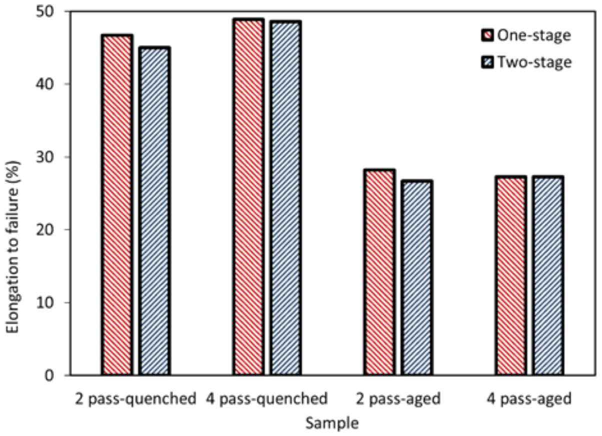
The values of yield strength (Y.S.), ultimate tensile strength (UTS) and elongation to failure (e_f) together with the hardness values of the samples in the as-solutionized state and in the as-aged state are shown in Figure 14. It is clear that the samples that experienced the two-stage solution treatment exhibited higher values of YS, UTS and hardness. In addition, elongation to failure, an important property of the alloy for structural applications, was not significantly reduced.



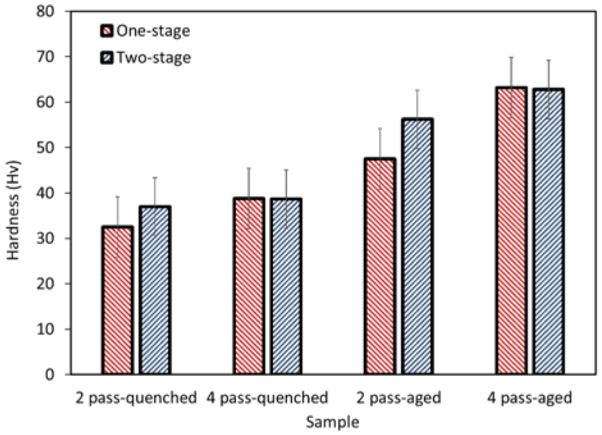
(a)



(b)



(c)



(d)

Figure 14- Tensile properties: (a) YS, (b) UTS, (c) e_f and (d) hardness of the samples in the as-solutionized state and in the as-aged state.

4 Conclusions

In this research, the routine one-stage solution treatment for the AA6063 alloy was replaced by a two-stage solution treatment, in order to avoid the occurrence of AGG to the cold-worked material and to achieve high mechanical properties. Based on the results obtained from this investigation, the following conclusions have been drawn.

1- Abnormal grain growth (AGG) indeed occurred to the samples during the one-stage solution treatment. Reducing soaking time could not provide a viable solution. Although the samples undergoing the two-stage solution treatment were soaked at the same temperature for the same time, no AGG occurred, because the stored deformation energy was largely consumed during the first soaking at the lower temperature.

2- Significant differences in hardness, yield strength and ultimate tensile strength were observed between the samples after the one-stage solution treatment and the two-stage solution treatment. Indeed, the samples after the two-stage solution treatment exhibited higher hardness and strength, with negligibly small reductions in elongation. Similar results were obtained for the samples in the as-solutionized state and in the as-aged state.

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