Microstructure and mechanical properties of an Al–Mg–Si tube processed by severe plastic deformation and subsequent annealing

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This study is aimed to realize evolution of microstructure and mechanical properties of aluminum 6061 alloy tube subjected to Severe Plastic Deformation (SPD) and subsequent annealing. For this purpose, the tube is initially processed by different passes of an SPD process called Tube Channel Pressing (TCP) and then subjected to a subsequent annealing at 473 °K for 2 h. Afterwards, tension test is used for the evaluation of mechanical properties while Electron Back-Scattered Diffraction (EBSD) equipped Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) are utilized for the micro-structural characterizations. Results show that the Continuous Static Recrystallization (CSRX) is the main restoration phenomenon during annealing of aluminum 6061 alloy, even after imposing a moderate plastic strain. For instance, CSRX has been observed during annealing treatment after imposing an equivalent plastic strain as low as 1. However, the used annealing treatment causes different micro-structural variations in specimens depending on the pass number of TCP. As an illustration, while the average grain size impressively decreases due to annealing of 1 pass TCPed specimen, it moderately increases after annealing of 5 passes TCPed specimen. This is due to development of a bimodal microstructure after 5 pass of TCP which leads to a different evolution of microstructure during successive annealing. It is also notable that TCPed and annealed specimens show higher strength and ductility compared with as TCPed specimens which is attributed to the occurrence of precipitation hardening besides restoration phenomenon during the annealing treatment.

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1. Introduction

One of the most common methods for grain refinement of materials is application of Severe Plastic Deformation (SPD) processes in which reduction of the grain size takes place by imposing intense plastic strains (e.g. equivalent strain of 1–100) without a significant change of outer dimensions. For instance, Very-Fine Grained (VFG) and Ultra-Fine Grained (UFG) materials can be produced using SPD method which their grain sizes are between 1 and 10 μm and less than 1 μm, respectively. Usually, a decrease of deformation temperature causes an increase of the strengthening rate and a decrease of the grain size reduction saturation limit. Therefore, SPD processes are often applied in cold and warm regimes [1–3].

As shown before, the grain refinement of metals subjected to a cold/warm SPD process \( T_{\text{deformation}} < 0.5 T_m \) occurs due to Continuous Dynamic Recrystallization (CDRX) [4]. In CDRX, the grain refinement takes place by propagation of Low Angle Boundaries (LABs) and following evolution of LABs to High Angle Boundaries (HABs) inside “parent grains” due to imposing intense plastic strain. Different mechanisms have been presented for this phenomenon as thoroughly discussed before. As an illustration, it is believed that CDRX in materials bearing high Stacking Fault Energy (SFE), such as pure and lowly alloyed aluminum, is mainly taken place by two mechanisms [3–5]:

(1) Multiplication and Migration of Dislocations (MMD): homogenous multiplication of dislocation as a result of deformation, migration and rearrangement of dislocations to form homos-shaped sub-grains (or LABs), rotation of these sub-grains to form main grains characterized by HABs.

(2) Intersection of Micro-Shear Bands (MSB): appearance of LABs due to propagation of Micro-Shear Bands (MSBs) as a result of deformation, evolution of LABs to HABs due to intersection of MSBs by further deformation.

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It is noteworthy that the IMSB usually results in a bimodal necklace shaped microstructure while the MMD results in a relatively homo-shaped microstructure as illustrated in Fig. 1.

Although a cold/warm SPD treatment can result in a reduction of the grain size, the kinetic of this phenomenon is relatively slow. For example, high strains in range of 8–12 are needed to develop an UFGed material by a cold/warm SPD treatment. In addition, the strain hardened materials obtained after a cold/warm SPD treatment reveal relatively low ductility [1,4]. Therefore, the cold/warm SPD of materials are often combined with subsequent annealing treatments which usually lead to occurrence of Static Recrystallization (SRX) [4,6]. SRX can occur either as Discontinuous SRX (DSRX) or Continuous SRX (CSRX) which are schematically illustrated in Fig. 2. As can be seen, DSRX occurs by nucleation and growth of new grains. In this phenomenon, a nucleus grows up by long range migration of its grain boundaries and forms a new grain bearing little dislocation density. Comparatively, CSRX takes place by initiation of LABs and their evolution to HABs to form new grains instead of old strain hardened grains. This occurs by migration and rearrangement of dislocations to form LABs and following evolution of LABs to HABs by further accumulation of dislocations in LABs [6]. Compared with those developed by DSRX, the dislocation density in grains developed by CSRX is relatively higher. This leads to higher inside misorientation of grains developed by CSRX. In addition, while DSRX results in equilibrium and sharp grain boundaries, CSRX results in ill-defined and wavy grain boundaries [4]. It is believed that CSRX takes place by annealing treatment after imposing intense strains while DSRX occurs by annealing after imposing relatively lower strains [4,6].

It is estimated that about 90% of aluminum extruded products are made from Al–Mg–Si alloys since these alloys have attractive characteristics such as the precipitation hardening capability and the excellent workability. Therefore, multiple studies have focused on SPD of these alloys [7–19]. However, the majority of these studies have considered Al–Mg–Si alloys in simple geometries (i.e. Billets and Sheets) and SPD of extruded Al–Mg–Si products, such as tubes, has been less studied. Recently, a new SPD process called Tube Channel Pressing (TCP) has been developed to process tubular geometries [9,10]. Since precipitation hardening can be activated during the annealing treatment of Al–Mg–Si alloys [7,12–15], the combination of TCP and post annealing of these alloys can be a good solution for development of tubes bearing an excellent strength and a good ductility. On the other hand, a few studies concentrated on the evolution of mechanical properties during annealing/aging treatments after SPD processes suggest that the effects of these treatments are strongly dependent on the imposed strain by SPD [15,20]. Therefore, this work aimed to study evolutions of microstructure and mechanical properties of an Al–Mg–Si tube processed by TCP and subsequent annealing.

2. Process, materials and methods

The schema of TCP is shown in Fig. 3. As can be seen, the tube passes in a bottleneck channel which results in a consecutive decrease and increase of its diameter. Additionally, the direction of curvature of tube wall is changed consequently during TCP.
Therefore, consecutive shear strains also impose during TCP. The average equivalent plastic strain imposed by each pass of TCP using the die characterized by \( R_{\text{die}} \) of 7.5 mm and \( \theta_{\text{die}} \) of 26° has been calculated about 1 by an FEM analysis [10].

An Al–Mg–Si alloy tube was received in extruded form. The inner and outer diameters of tube were 19 mm and 26 mm, respectively. The chemical composition of tube was Al–1.01 Mg–0.49Si–0.31Cu–0.24Fe–0.06Cr wt% which is identical to the aluminum 6061 alloy. 75 mm long specimens were cut from as received tube and subjected to solution treatment at 803 K for 1 h. This solution treatment causes dissolution of almost all precipitations since after this treatment, no evidence of any precipitation was detected by X-Ray diffraction [7,21,22]. After solution treatment, different specimens were subjected to 1, 3 and 5 passes of TCP at the room temperature. These specimens are called 1TCP, 3TCP and 5TCP, respectively. After processing by TCP, a group of specimens were subjected to an annealing treatment at 473 K (about 0.5\( T_{\text{m}} \)) for 2 h and then immediately quenched in room temperature water. These specimens are called 1TCPA, 3TCPA and 5TCPA, respectively. Tension samples were machined parallel to longitudinal direction of specimen as illustrated previously [21,23]. The strength of specimens was obtained by tension tests at the room temperature using the strain rate of \( 5 \times 10^{-4} \) s\(^{-1} \).

Transmission Electron Microscopy (TEM) samples were prepared by mechanical polishing to 100 \( \mu \)m thickness and subsequent jet-polishing in a 75% \( \text{CH}_3\text{OH} \)–25% \( \text{HNO}_3 \) solution at –30 °C for 5–15 min. The TEM studies were accomplished by at
least three different samples from each studied specimen using JEM-2100F machine adapted in the acceleration voltage of 200 kV. Scanning Electron Microscopy (SEM) samples were prepared by ion polishing using the IB-9010 cross section polisher to obtain Electron Back Scattering Diffraction (EBSD) maps. These maps were achieved by JSM-7001F machine adapted at the acceleration voltage of 15 kV and the INCA 4.09 software. The mapping was repeated at least three times for each specimen to obtain relatively accurate results. The step sizes of EBSD measurement were selected between 0.5 and 0.1 μm. The cut off angle for definition of HABs in EBSD maps was considered as 5° since it has been shown that the appearance of grain boundaries with angle higher than 5° in aluminum results in Hall–Pitch strengthening [24,25].

3. Results

Fig. 4(a)–(d) compares EBSD maps of the specimens subjected to different passes of TCP. As can be seen in Fig. 4(a), the as received tube has equiaxed Coarse Grains (CG) with an average grain size of 35 μm. As shown in Fig. 4(b), few UFGs have appeared after the first pass of TCP which implies a limited alteration of the
microstructure. Imposing of 3 passes of TCP results in a mixed microstructure which consists of UFGs, VFGs and CGs as illustrated in Fig. 4(c). After 5 passes of TCP, the microstructure mainly consists of UFGs and VFGs as shown in Fig. 4(d). Comparing Fig. 4(a)–(d), it is obvious that the processing by TCP causes a development of a bimodal necklace shaped microstructure as discussed later.

Annealing of 1TCP specimen results in arise of finer grains as shown in Fig. 4(e). In 1TCPA specimen, two types of grains can be traced: UFGs appeared during TCP and VFGs developed during subsequent annealing treatment. These VFGs substitute initial coarse strain hardened grains and are characterized by ill-defined wavy boundaries as can be seen in Fig. 4(e). As shown here, there is a relatively high misorientation inside these grains which indicates their high dislocation densities. Similar microstructures can be seen for 3TCPA and 5TCPA specimens as illustrated in Fig. 4(f) and (g). Nonetheless, comparing Fig. 4(d) and (g), it is obvious that the annealing treatment has impressively lowered fraction of finer grains (e.g. UFGs) in 5TCPA specimen. In addition, VFGs appeared in 5TCPA specimen are relatively coarser than VFGs of 5TCP specimen. This indicates the occurrence of grain growth in this specimen as discussed later.

Fig. 5(a) compares the average grain size of specimens before and after annealing treatment. As illustrated here, the annealing treatment after 1 pass of TCP causes a remarkable decrease of the grain size. Despite so, the grain size is moderately increased after annealing of 5TCP specimen. Fig. 5(b) shows the HABs fraction for specimens before and after annealing treatment. As can be seen, the HABs fraction is impressively increased after annealing of 1TCP specimen. Despite so, a notable decrease of HABs fraction can be observed after annealing of 5TCP specimen. Despite so, a notable decrease of HABs fraction can be observed after annealing of 5TCP specimen. Fig. 5(c)–(e) compares the distribution of grain size in xTCP and xTCPA specimens. As can be seen in Fig. 5(c), the annealing treatment after 1 pass of TCP
causes a relative decrease in fraction area of coarser grains (e.g. grains greater than 30 μm) in benefit of increase of fraction area of finer grains (i.e. grains in range of 0–3 μm). This is due to occurrence of recrystallization phenomenon. Comparatively, the annealing treatment after 5 pass of TCP causes a decrease of fraction area of finer grains and an increase of fraction area of coarser grains which indicates occurrence of the grain growth during annealing.

Fig. 6(a)–(f) compares the microstructures of specimens processed by TCP and subsequent annealing. Collating Fig. 6(a) and (c), it is obvious that the annealing treatment after 1 pass of TCP causes development of finer grains which are characterized by ill-defined wavy boundaries. In comparison, the evolution of microstructure during annealing of 5TCP specimen is relatively different as shown in Fig. 6(b) and (d). At first, the grain boundaries are sharper in 5TCPA specimen in comparison of 1TCPA specimen. This is due to imposing of a larger strain which promotes recrystallization phenomenon. Secondly, while UFGs developed by TCP can be easily found in 5TCP specimen, they can hardly be traced in 5TCPA specimen. This illustrates growth of grains during annealing of 5TCP specimen which is mentioned before. Besides evolution of grains, the annealing treatment results in

Fig. 6. Microstructures of different specimens: (a) 1TCP, (b) 5TCP, (c) 1TCPA and (d) 5TCPA; Appearance of precipitations in: (e) 1TCPA specimen and (f) 5TCPA specimen.
development of precipitations as shown in Fig. 6(e) and (f). As thoroughly discussed before [7,22,26], it is believed that the consequence of precipitation in an Al–Mg–Si alloy is:

GP – I Zone ∆→ GP – II Zone (β′′) → β’ → β

It is well-known that the morphology of GP-I zone is spherical while the morphology of β’, β’’ and β are needle-like (elliptical), rod-like and platelet-like, respectively. As can be seen, precipitations appeared in 1TCPA and 5TCPA specimens have an elliptical morphology which has been related to β’’ precipitations [7,22,26]. In addition, the size of precipitation appeared in 5TCPA specimen is coarser than that appeared in 1TCPA specimen. This is attributed to the remarkable increase of diffusion rate by increase of imposed plastic strain in SPDed materials as discussed before [27–30]. The emergence of precipitations during annealing treatment results in an improvement of strength as discussed later.

Fig. 7 compares evolutions of the strength and ductility of specimens after processing by the TCP and subsequent annealing. As can be seen, yield strength of 310–380 MPa and fracture strain of 17–25% is obtained by the used treatment depending on the TCP pass number. Comparatively, the strength and the ductility of similar Al–Mg–Si alloys treated by other SPD and heat treatment procedures are reported in ranges of 240–420 MPa and 5–20%, respectively [7–19]. This denotes that the treatment used in this work has shown a significant capability to improve the strength and the ductility of tube.

4. Discussion

From what told above, it can be inferred that TCP results in development of a bimodal necklace-shaped microstructure similar to what illustrated in Fig. 1. As discussed before, this microstructure arises due to occurrence of IMSB promoted CDRX [3,4,9]. In general, the IMSB mechanism leads to development of two groups of grains: finer grains (i.e. UFGs) and coarser grains (i.e. VFGs and CGs) similar to what shown in Fig. 4(a)–(d). In comparison, the microstructures of TCPed and annealed specimens consist of finer grains developed during TCP and coarser grains developed during TCP and/or annealing as can be seen in Fig. 4(e)–(g). As shown here, coarser grains developed during annealing are characterized by ill-defined wavy boundaries and bear a high dislocation density. These trends are similar to what shown in Fig. 2(b) for the evolution of microstructure by CSRX which indicates occurrence of this phenomenon during applied annealing treatment for used alloy. It is notable that UFGs developed during an SPD process bear low inside dislocation density [4]. Regarding this, one may propose that these UFGs may act as nucleus for initiation of DSRX during subsequent annealing of Al–Mg–Si alloys subjected to SPD. Despite this, results of this work decline this idea since the microstructure of TCPA specimens represent occurrence of CSRX rather than DSRX as explained in discussions for Fig. 4(e)–(g). Therefore, it can be demonstrated that the CSRX is the predominant restoration phenomenon of an Al–Mg–Si alloy subjected to a moderate plastic strain – as low as 1 – during subsequent annealing. Comparatively, occurrence of CSRX is reported during annealing of pure copper and stainless steels after imposing strains greater than 3 [4]. This difference can be explained by high SFE of aluminum compared with those materials which accelerates its dislocation mobility [6,13,14].

As mentioned in explanations of Fig. 5, while the decrease of average grain size and increase of HABs fraction after annealing of 1TCP specimen occurs due to CSRX, the increase of average grain size and decrease of HABs fraction after annealing of 5TCP specimen occurs due to grain growth phenomenon. This is related to
higher progression of CDRX in 5TCP specimen during TCP which makes it susceptible for subsequent grain growth during annealing. Compared with 1TCP specimen, STCP specimen has been affected by more severe CDRX which causes much progression of recrystallization and higher fraction of UFGs. Besides this, STCP specimen bears greater stored energy and higher rate of diffusion due to more intense plastic deformation [27–30]. Therefore, the affinity for grain growth during subsequent annealing of STCP specimen is higher than that of 1TCP specimen which causes rapid elimination of finer grains (i.e. UFGs) in the presence of coarse grains. It is also notable that the upper bound of grain size in STCPA specimen is 30 μm as shown in Fig. 5(d) which is much greater than 9 μm related to STCP specimen. This represents an extensively accelerated and abnormal grain growth in a group of grains which is not acceptable at the used annealing temperature. In fact, limited growth of microstructure has been reported during annealing of the used alloy in this temperature (473 K) in previous studies [13,31,32]. In comparison, the abnormal grain growth during annealing after 5 passes ofTCP is related to development of a bimodal microstructure during TCP which accelerates the growth of grains due to a great driving force for elimination of finer grains (i.e. UFGs) in the microstructure. In addition, since the fragmentation of initial microstructure through TCP has been occurred by IMSB mechanism, the elimination of finer grains developed by IMSB results in viscosity of coarser grains to each other (see Fig. 1). These coarser grains may have a similar crystallographic orientation since they may be derived from one parent grain. Therefore, the elimination of finer grains causes a reduction of HABs and abnormal increase of grain size due to a probable coalescence of coarser grains as illustrated in Figs. 4 and 5.

As illustrated in Fig. 7(a), 2 h of annealing of TCPed specimens at 473 K results in a moderate increase of their yield strength. This can be explained by activation of age hardening during annealing as shown before [7,12–15]. However, little changes have been previously obtained for strength of TCPed specimens after 20 min. of annealing in similar temperature [21]. This has been explained by decrease of dislocation density besides arise of pre-β′ precipitations which results in a balance between strengthening effect of precipitations and softening effect of restoration [21]. Collating with those results, the moderate strength increase of TCPed specimen after 2 h of annealing is related to incidence of semi-coherent β′ precipitations which causes more strengthening effect in comparison of coherent pre-β′ precipitations [19,26]. In addition, the annealing strengthening effect is decreased by increase of TCP pass number as shown in Fig. 7(a). This is related to incidence of more coarse precipitations during annealing of a specimen subjected to higher TCP pass numbers as previously illustrated in Fig. 6(e) and (f). Note that coarsening of precipitations reduces their strengthening effect due to increase of their free distance [14]. Beside moderate strength increase, the elongation is impressively increased by annealing treatment after TCP as illustrated in Fig. 7(b) which can be explained by restoration effect of annealing. It is also noteworthy that the fracture strains of TCPA specimens are impressively higher than that of the solution treated-annealed/aged specimen. This is mainly due to increase of the strain accommodated after necking point in TCPA specimens as illustrated in Fig. 7(c). As can be seen, the necking strains of TCPA specimens are almost equal to necking strain of solution treated-annealed/aged specimen. However, TCPA specimens bear more extensive strain after necking point which results in their higher ductility. This can be explained by the lower grain size of TCPA specimens which increases their strain rate sensitivity and decreases the occurrence of fracture after necking point [33–35].

5. Conclusions

Considering the presented results, it can be concluded that:

1. The development of a bimodal microstructure during TCP results in occurrence of different trends during subsequent annealing of TCPed specimens.
2. Continuous static recrystallization is the main restoration phenomenon during annealing of the aluminum 6061 alloy at 473 K after imposing a moderate plastic strain as low as 1.
3. The average grain size is impressively decreased by annealing after 1 pass ofTCP due to occurrence of continuous static recrystallization. Despite so, annealing after 5 passes ofTCP causes a moderate increase of the average grain size which is related to elimination of finer grains by occurrence of grain growth during annealing.
4. Since the restoration and the precipitation hardening occur together, both of the strength and ductility of the aluminum 6061 alloy improve by 2 h of annealing at 473 K after TCP.

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