Grain Refinement and Mechanical Behavior of the Al Alloy, Subjected to the New SPD Technique

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The paper focuses on producing of ultrafine-grained (UFG) structure in the Al 6061 alloy by a new severe plastic deformation (SPD) technique, developed recently in our laboratory, namely equal channel angular pressing with parallel channels (ECAP-PC). The evolution of microstructure at ECAP-PC was examined and was proved that the alloy becomes of a homogenous UFG structure after 4 passes. Such a structure increases essentially the alloy’s mechanical properties, specifically strength and ductility. The advantages of this new technique in producing of UFG alloys over conventional ECAP are considered and discussed as well. [doi:10.2320/matertrans.MD200821]

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1. Introduction

Fabrication of ultrafine-grained (UFG) metals and alloys by severe plastic deformation (SPD) processing has become a well-known direction in modern materials science.1–3) These works started in the early 90-s with two techniques: equal channel angular pressing (ECAP) and high pressure torsion (HPT),4) have been developed intensively last decade in connection with creation of principles of materials nano-structuring for advanced properties and elaboration of more effective SPD techniques.

Conventional ECAP technique, widely used to produce UFG structure in metals and alloys is currently applied mainly as laboratory-based SPD method as it has some limitations, which restrict its industrial application. These restrictions are due to labor-intensity and low efficiency of the method, because a large number (8–12) of passes is required for a UFG structure formation and only small amount (less than 60%) of the material is subjected to homogenous shear straining in the bullet.5) The new SPD technique-equal channel angular pressing with parallel channels (ECAP-PC),6,7) newly developed in our laboratory is able to reduce a number of restrictions for the conventional ECAP. Computer simulation and experiments applied to Cu and Ti as model materials have shown, that UFG structure is produced more effectively by ECAP-PC, due to more advanced strain and homogeneity rates.5,7)

The given paper reports the first successful application of the new SPD technique to commercial Al alloy with the aim of producing an UFG structure and improving its mechanical properties. The experiment was conducted on 6061 Al alloy. This alloy is one of the commercial aluminum alloys widely used in aerospace and automobile industries. Advantages of the new method versus conventional ECAP are being discussed as well.

2. Experimental Procedure

A commercial 6061 aluminum alloy (0.80Mg-0.41Si-0.12Cu-0.03Mn-0.02Ti-0.22Fe-balance Al (mass%)) was used in a cast condition. The alloy under investigation was characterized by lower level of alloying elements (Si and Mg), comparing to traditional Al 6061 alloy.8) Before ECAP-PC treatment, initial billets were subjected to solid solution treatment for 5 h at 530°C with the following water quenching.

The principles of ECAP with PC are shown in Fig. 1.5,7) A distinctive feature of the ECAP-PC is that during a single processing pass, two distinct shearing events take place: shear in two deformation zones subsequently corresponding to two subsequent channels intersections in the die-set (Fig. 1(a), (b)).

The value of the displacement between the two channels, K, and the angle of intersection of the two channels Φ are the main parameters of the die geometry, which influence both the flow pattern and the strain-stress state of the ECAP process. It has been established by computer simulation, that the optimal value of these parameters leading to the largest strain homogeneity are Φ = 100° and K = d, where d is the channel diameter.5,7) Under these conditions, the accumulate strain for one pass is approximately equal to 2. The simulation results have been confirmed experimentally using a grid method. Furthermore, the studies showed that the deformation pattern realized in ECAP-PC die-set optimal geometry makes the structure homogeneous along the length of the bulk sample including up to the ends (the material utilization ratio is higher than 90%).

Billets of 6061 Al alloy with the diameter of 18 mm and 100 mm length were subjected to ECAP-PC at 100°C, since the temperature provides significant grain refinement, preserving the facility for its further strengthening by subsequent aging at 160–175°C.

Vickers hardness (HV) was measured using a Micromet-5101 microindentation tester with a load of 200 g for 15 s. In order to receive reliable results, each sample was measured more than 10 times.

Tensile tests at room temperature were performed on Instron 1185 at a strain rate of 5 × 10^-4 s^-1. Mechanical properties were measured by at least 2 specimens with gage length 15 mm and diameter of 3 mm. Tensile specimens were cut of parallel to the longitudinal axis of the ECAP-processed material.

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The structural characterization by TEM was performed in JEM-100B and JEOL-2000 EX electron microscopes using dark and bright fields. Average grain size was estimated from more than 250 grains measurement.

3. Results and Discussion

Observations by TEM showed that mainly subgrain structure of a lamellar type with clear orientation to shear plane was formed after ECAP-PC thorough one pass at 100°C (Fig. 2(a)). The mean cross subgrain size comprised 400 nm and longitudinal one made up 1200 nm correspondingly. Electron diffraction patterns prove subgrain structure formation with mainly low-angle grain boundary misorientations (Fig. 2(b)). After ECAP-PC through the second pass at the same temperature the structure changed significantly. (Fig. 3). Fragmentation by means of cross-subboundary formation can be seen in elongated subgrains formed after ECAP-PC through 1 pass (Fig. 3(a), (b)). Separate areas (with a volume fraction of around ~ 30%) of equi-axed grains of 500 nm are also observed in the structure along with subgrain fragmentation (Fig. 3(c), (d)).

The electron diffraction pattern (Fig. 3(f)) represents numerous spots distributed uniformly over the circles. This testifies to formation of the UFG structure at the areas of a granular type with high-angle grain boundaries at the areas.

The UFG structure formed after ECAP-PC through 4 passes is also of equiaxed (Fig. 4). Besides, secondary phase dispersive precipitates of less than 10 nm in size were found in the alloy UFG structure after ECAP-PC (Fig. 4, A and B areas). According to the results9,10) dealing with 6061 Al alloy processing by conventional ECAP, these dispersive precipitates represent Mg2Si particles. These precipitates testify to the UFG structure formation being accompanied by dynamic aging at ECAP-PC through 4 passes. As the precipitates were not found after the ECAP-PC through 1 and 2 passes, we can suggest that they are absent in these states, or their size was quite small and required investigations by high-resolution electron microscopy. It should be also noted that there were more lattice dislocations in the
The Vickers hardness (HV) of the initial billets after solid solution treatment was 410 ± 18 MPa. ECAP-PC processing increases the rates up to 1155 ± 24 MPa and to 1220 ± 27 MPa after 1 and 2 passes correspondingly. However, the hardness values decreased to 1100 ± 21 MPa after ECAP-PC through 4 passes. Mechanical properties of 6061 alloy after ECAP-PC through 1, 2 and 4 passes are presented in Fig. 5 and Table 1. The data can be compared with that of the same alloy after conventional T6 treatment (quenching at 535°C and subsequent artificial aging at 160°C for 12 hours).

The results correlate closely to the evolution of the microstructure revealed (Fig. 2–4). The formed substructure (Fig. 2) with a high density of lattice dislocations provides significant increase in strength after ECAP-PC through 1 pass, but its ductility is low enough and elongation to failure comprised ~ 6.5%. The formation of the UFG structure of a combined type containing grains and subgrains (Fig. 3) in the billets after ECAP-PC through 2 passes provides some increase in both strength and ductility up to ~ 8% (Table 1).

At the same time the homogeneous UFG structure (Fig. 4) formed after ECAP-PC through 4 passes leads to insignificant loss in strength. However, its ductility increases by two times and its elongation reaches ~ 20%. Herewith, the UFG alloy demonstrates considerable uniform elongation which makes up ~ 9%. Such a high value, which comprises ~ 40–45% from total elongation, shows that the alloy with UFG structure has a potential to display enhanced fatigue.5,11)

Thus, the analysis of structural changes in the alloy 6061 after ECAP-PC at 100°C proves that the ECAP-PC technique makes it possible to produce a homogeneous UFG structure in a cast alloy after 4 passes in comparison with 8–10 passes in conventional ECAP,5,9) in other words, using of ECAP-PC can decrease the number of passes in 2–3 times under nearly the same processing conditions. Another advantage of the pressing is significant increase in the volume of UFG structure in a billet processed: about 85–90% compared to 50–60% after conventional ECAP. This can be of special interest for the process industrial application.

Regarding UFG structure formation in the alloy, an important feature of ECAP-PC is producing of practically equiaxed UFG structure already after 2 passes (Fig. 3),
though the grains observed after the first pass were of elongated laminar structure (Fig. 2(a)). Gradual transformation of the grains to equiaxed ones is typical also of conventional ECAP. At the same time, the present work proves the connection of this transformation with the formation of low-angle subgrain boundaries inside the elongated grains (Fig. 3(a)) and their following arrangement to high angle boundaries of the equiaxed UFG structure. Similar evolution of nanostructure during SPD processing has been discussed elsewhere.12

![Fig. 4](image.png) Fig. 4 Microstructure of the alloy after ECAP-PC through 4 passes in the longitudinal section of the sample (a), (b), in the cross-section of the sample (disperse particles of Mg₂Si phase precipitated during the processing-areas A and B) (d), SAED pattern (c).

![Fig. 5](image.png) Fig. 5 The engineering stress-strain curves of the 6061 Al alloy at room temperature after ECAP-PC processing and convention heat-treatment (T6).

![Table 1](image.png) Table 1 Mechanical properties of the 6061 Al alloy at room temperature after ECAP-PC at 100°C.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>UTS, (MPa)</th>
<th>YS, (MPa)</th>
<th>El., (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ECAP-PC at 100°C, 1 pass</td>
<td>370</td>
<td>335</td>
<td>6.5</td>
</tr>
<tr>
<td>ECAP-PC at 100°C, 2 passes</td>
<td>380</td>
<td>350</td>
<td>8.0</td>
</tr>
<tr>
<td>ECAP-PC at 100°C, 4 passes</td>
<td>345</td>
<td>305</td>
<td>20.0</td>
</tr>
<tr>
<td>T6</td>
<td>230</td>
<td>178</td>
<td>30.0</td>
</tr>
</tbody>
</table>
One more effect revealed in the alloy after 4 passes is the formation of highly-disperse precipitations of the second phase, obviously, the particle of Mg$_2$Si phase. The formation of such particles evidences dynamic aging, which was observed recently in 6061 alloy after conventional ECAP.\cite{1,2,9,10} However, this time the aging occurs at a low temperature (100°C) and aging time less than 10 minutes, which is considerably lower, than 175°C, 8 h of the peak aging condition (T6) for a 6061 alloy.\cite{10} Such an acceleration of aging kinetics can result from high vacancy concentration during processing. Recent \textit{in situ} HPT experiments in synchrotron beamline showed\cite{13} that excess vacancy concentration can reach $10^{-3}$, which is near to equilibrium concentration at melting temperature point. Besides, 3D atom probe applied to the alloy showed the formation of alloying elements segregations after HPT even at room temperature.\cite{14} Such segregations appear to be the second phase precipitations at the subsequent deformation. Thus, aging processes are more rapid at ECAP-PC, which can influence the alloy’s mechanical behavior.

It is well-known, that ECAP processing significantly increases the strength, but reduces ductility. The Al 6061 alloy after ECAP-PC through 1 and 2 passes behaved in accordance with this rule. However, it showed considerable increase in ductility with the small reduction of strength (to 30–40 MPa) after ECAP-PC through 4 passes. Such extraordinary combination of high strength and high ductility of UFG Al alloy was also observed recently.\cite{5,15–17} This attractive mechanical behavior can be caused by either formation of UFG structure with high-angle grain boundaries capable to grain boundary sliding,\cite{16} or difficulty in strain localization due to the presence of disperse particles.\cite{17} We are planning to study the phenomenon in detail in our ongoing research.

Conclusions:

1) Using ECAP-PC we could successfully produce UFG structure in Al 6061 alloy. This new technique has a number of advantages over conventional ECAP for grain refinement. These advantages attribute to gain in material amount in the rod.

2) The UFG structure formation during the processing of the alloy is associated with transformation of low-angle boundaries to high-angle grain boundaries. The formation is also accompanied by acceleration of dynamic aging processes.

3) The 6061 Al alloy after ECAP-PC through 4 passes showed combination of high strength and ductility, which is of special interest for its advanced application.

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REFERENCES