Ultra Fine Microstructure and Properties Formation of EN AW 6082 alloy

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The influence of heat treatment conditions and severe plastic deformations (SPD) by ECAP on structural and mechanical properties development of EN AW 6082 aluminium alloy was investigated. The hardness and mechanical properties in dependence on two types of solution heat treatment (TSA=400°C+slow cooling and TSA = 550°C +quenching), natural or artificial ageing treatment (room temperature, 100°C, 170 °C), several annealing temperatures (T A=100 - 300°C) and ageing or annealing times after SPD were tested. The best properties for EN AW 6082 material were achieved after processing by (T A=550°C/1,5h + water quenching + 3xECAP/20°C + T A=100°C/30h) on level: 0.2%YS=408MPa, UTS=427MPa, El.=20.1%, Re.=29.3%, KCVRT=16.5J.cm⁻².

Key Words: EN AW 6082, Heat Treatment Conditions, Hardness, Mechanical Properties, Microstructure

1. Introduction

The middle-strength AlMgSiMn alloys are suitable for different miscellaneous structural applications in the building, automotive and aircraft industries, due to their strong modification of strength induced by precipitation phenomena which is accompanied by a low density, good corrosion properties and good weldability. The most useful aluminium alloys as stressed structural members are EN AW 6082 (medium strength) and EN AW 7075 (very high strength). EN AW 6082 alloy uses an excess amount of silicon (Si/Mg > 1) to increase age hardening response and addition of manganese (typically 0.7 wt. %) to control grain size, while Fe is a natural impurity (~ 0.2 wt. %)¹. A wide variety of Fe-containing intermetallics are formed between aluminium dendrites during casting of this alloy²⁻⁴. During necessary homogenization treatment of as cast billets several processes take place such as transformation of β-Al₃FeSi to α-Al₁₂(FeMn)₃Si intermetallics, which is important for alloy ductility improvement⁵. Dissolution of β-Mg₃Si during homogenization is also important since it will give maximum age hardening potential for the extruded product²⁻⁴⁵.

In aluminium alloys, it is difficult to obtain fine structures with grain size diameter <10μm using the conventional processing by plastic deformation with the following recrystallization or thermo-mechanical treatments. This is due to the physical properties of these alloys⁶. Despite this fact, it is possible to obtain ultra-fine grain structures of aluminium alloys with grain size <1 μm by applying bulk deformation based on SPD processes⁷⁻⁹. SPD is currently a subject of considerable attention, because it allows to obtain Al-based alloy structures at nanometre level¹⁰. ECAP (equal channel angular pressing)¹¹⁻¹³ is an effective SPD method introducing intensive plastic deformations to the sample with repeated pressing process. A possible mechanism of high-angle boundary nanograins evolution consists of formation of cell structure, subgrains that transform with the increase of deformation into nanograins with high-angle misorientation¹⁴⁻¹⁷. The result of such grains refinement is first of all the improvement in mechanical properties¹⁸⁻²⁰ and also the density of lattice defects in the solid solution of Al-based alloys increases markedly and thus accelerates the precipitation process of strengthening particles during the subsequent ageing. Some other possibility for strength properties increase can be reached by these combinations: solid solution treatment + quenching or slow cooling + ECAP + age hardening. Some authors¹⁴,²¹ studied this combination on the middle and high strength aluminium alloys with positive effect on mechanical properties increasing. Paper aim is increasing Al properties by structural formation via SPD not with arrangement of chemical composition.

2. Experimental material and methods

The investigation has been carried out on the commercial aluminium alloy EN AW 6082. The chemical composition of the analyzed alloy is indicated in Table 1.

<table>
<thead>
<tr>
<th>Mg</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
<th>Cr</th>
<th>Zn</th>
<th>Cu</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.87</td>
<td>0.90</td>
<td>0.85</td>
<td>0.19</td>
<td>0.09</td>
<td>0.03</td>
<td>0.08</td>
<td>bal</td>
</tr>
</tbody>
</table>

The extrusion bars industrially processed in T6 temper were used like initial state (IS). The influence of
solution annealing (SA) temperature on hardness after water quenching (WQ), kinetics of ageing and microstructures after ageing was investigated. The experimental schemes are shown in Figs. 1-3.

The samples in the experimental schedule after Fig. 1. were heated to a wide range of SA temperature from 530 to 600°C and holding time 1.5h in vacuum with the following rapid water quenching to room temperature. In the experiment after Fig. 2. samples were vacuum reheated on the constant SA temperature 550°C during 1.5h with the following rapid water quenching to room temperature and then three ECAP passes at room temperature were made. The two different routes were used for the next sample processing: reheating on two SA temperatures 100°C and 170°C and several times of artificial ageing process, natural ageing (NA) at room temperature during 800h.

The samples in an experimental schedule after Fig. 3. were annealed at the constant SA temperature 400°C during 2,5h with the following slow furnace cooling (100°C/h) to room temperature and then four ECAP passes at room temperature were made. The six various annealing (AA) temperatures and several times were used for this ECAPed state. The ECAP process was performed in a die with the parameters: Ψ=90° a Ψ=37°. Repetitive pressing of specimens of size Ø 10 mm x 80 mm was attempted in the ECAP die at room temperature using C route. The influence of temperature – time and deformation regimes on the central part of the sample was analyzed by: the Vickers hardness measured under loading 98 N (HV10), the tensile test on short specimens (D₀xL₀=5x10mm with the deformation rate 2.5.10⁻⁴s⁻¹), the impact toughness test on the longitudinal sample with V-notch direction (1mm in width and 1mm in depth, h x b x l=4 x 8 x 55mm) at room temperature, light and TEM microscope.

3. Results and discussion

3.1. Hardness

3.1.1. Solution annealing

Hardness of the sample in the initial state (industrial hot extrusion and temper state T6) was 100HV. Hardness after reheating on SA temperature with the following WQ decreased to the values in the range from 63.4 to 68.9HV, as Fig. 4. 221 shows.

This alloy softening is a result of the solid solution recrystallization process and dissolution of Mg2Si precipitates responsible for strengthening of the studied alloy in the initial state. Subsequently, if the SA temperature was increasing from 530°C to 560°C, the hardness of quenched states increased, too. The hardness was increased, because the substitute strengthening of solid solution through the dissolution of accumulative amount of undissolved Mg2Si particles occurred during the SA process. The linear increase of the hardness value
in dependence on the SA temperature was also presented in [23]. The hardness at the SA temperatures in the interval from 560 to 590°C was approximately constant (~68.7HV). The constant hardness resulted from the structural changes having opposition effects on hardness. The growth of microscopic size and area portion of the coarse intermetallic particles of Al(FeMn)Si type evoked by the SA at temperature in the range (560 - 590°C) was observed microscopically [23] . This modification of coarse intermetallic particles occurred through the dissolution and coagulation of the fine intermetallic particles. The absence of these fine particles, which retard the grain growth, probably evoked the solid solution grain growth at high temperature of SA (>560°C) and then the hardness decreased. On the contrary, this hardness decrease was compensated, because the area portion of hard intermetallic particles increased during the SA at these higher temperatures. The local melting observed in the microstructure after the SA at 600°C resulted in considerable decrease of hardness value (60.2HV). A desirable SA temperature 550 °C was determined from the graphical dependence.

3.1.2. Solution annealed + SC + ECAP + aged states

The samples after SA at 400°C/2.5h were slowly cooled in furnace at cooling rate v =100°C/h, ECAPed at room temperature by 4 passes and annealed. The hardness dependence on treatment conditions is given in Fig. 5.

![Graph showing hardness vs. annealing time for different temperatures](image)

Fig. 5. Hardness dependence on annealing conditions. ([400°C/2.5h]+SC[100°C/h]+4xECAP)

Post ECAP hardness measured immediately after process without application of annealing was HV10=72.9. If ECAP process was accompanied by annealing treatment, the following results were obtained:
- for annealing temperatures 150°C, raising annealing time incurred not any important hardness increasing (a state is distinguished from a structural stability)
- for additional annealing temperatures (200-300°C), hardness was decreased with raising of annealing temperature and time (states are distinguished from interaction between structural recovery until to partial recrystallization and solution and coarsening of precipitates).

3.1.3. Solution annealed + WQ+ ECAP + aged states

The samples after SA at 550°C/1.5h were water quenched, ECAPed by three passes at room temperature and artificially aged at two temperatures (100°C and 170°C) for some times. The hardness dependence on treatment conditions is given in Fig. 6. Post ECAP hardness measured immediately after process without ageing application was HV10=130.3. If ECAP process was accompanied by artificial ageing treatment, the following results were obtained:
- maximal hardness HV10=141.7 was achieved with artificial ageing state T_{AA}=100°C /30h which is due to precipitation hardening probably of B' precipitates (hardness is descend after time 30h for precipitate coarsening)
- very similar hardness level HV10=139.5 in comparison with previous state was achieved by natural ageing state (20°C/800h) with precipitation hardening
- increasing of artificial ageing temperature on T_{AA}=170°C incurred hardness decreasing with rising time due to recovery of severely deformed microstructure.

![Graph showing hardness vs. artificial ageing time for different temperature conditions](image)

Fig. 6. Hardness dependence on artificial ageing conditions. ([550°C/1.5h]+WQ+3xECAP)

3.2. Tensile tests

3.2.1. Solution annealed and ECAP-ed states

The samples after SA at 400°C/2.5h were slowly cooled in furnace at cooling rate v =100°C/h and ECAPed at room temperature by some passes. The stress – strain curves from tensile tests were made as shown in Fig. 7.

Yield strength and tensile strength increased after ECAP deformations. The biggest stress growth was obtained after 2st ECAP pass. Maximal level of YS and
TS was achieved after 4th pass at which minimal value difference was observed between 3rd and 4th ECAP pass. Tensile ductility of ECAPed states was decreasing.

3.2.2. Solution annealed + WQ + ECAP + aged states

The samples were water quenched after SA at 550°C/1.5h, then ECAPed by three passes at room temperature and naturally (800h) or artificially (100°C/30h) aged. Comparison of mechanical properties determined for industrially processed - initial state (hot extrusion and artificial ageing) and ECAPed states is provided by the values of strength and tensile ductility, which are presented in Table 2. The tensile stress-strain curves of analyzed alloy states are shown in Fig. 8. Yield strength and tensile strength of the ECAPed and naturally aged (800h) analyzed alloy are higher (0.2% YS: +19.7 %; UTS: + 8.6%) than those of the industrially processed initial state (extruded and artificially aged) of analyzed alloy, but the tensile ductility of ECAPed and naturally aged alloy is lower (El.: -6.6%; Re.: - 20.4%). However, the ECAP-ed and artificially aged alloy (30h at 100°C) is even notably stronger (UTS: +2.2 %) than the naturally aged alloy after the repetitive ECAP passes. In addition, when the artificial ageing of ECAP-ed alloy replaced the natural ageing, the tensile elongation is improved from 13 % to 20.1%. This increased value of the tensile elongation is even little higher then that obtained for initial state of analyzed alloy. The increase in strength of the ECAP-ed and naturally aged alloy was first of all the consequence of the more expressive strain hardening of the solid solution by the ECAP process at room temperature in comparison with the conventional industrial process of hot extrusion and artificial ageing. The application of artificial ageing treatment after ECAP process improves the ductility and the strength because the hardening effect by the expected sequence Mg2Si-precipitation dominates the softening effect by microstructure low recovery and relaxation of internal stress\textsuperscript{24, 25}. This statement is also confirmed by comparison of the tensile stress - strain curves obtained for initial and ECAPed states, which show higher uniform deformation during tensile tests of specimens prepared from the initial or ECAP-ed and artificially aged state in comparison with that obtained by ECAP and natural ageing.

![Fig. 7. Stress - strain curves after SA, SA + ECAP and slow cooling from SA temperature.](image)

3.3. Impact test

Values of the absorbed energy during impact test of the analyzed alloy states are summarized in Table 3. for treatment conditions: SA at 550°C/1.5h-WQ, ECAP-ed by three passes at room temperature and naturally aged (800h) or artificially aged (100°C/30h).

![Fig. 8. Tensile stress-strain curves for the analyzed states.](image)

<table>
<thead>
<tr>
<th>alloy state</th>
<th>absorbed energy [J.cm(^{-2})]</th>
</tr>
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<tbody>
<tr>
<td>initial state</td>
<td>25.1</td>
</tr>
<tr>
<td>quenched</td>
<td>54.8*</td>
</tr>
<tr>
<td>ECAPed + naturally aged</td>
<td>12.5</td>
</tr>
<tr>
<td>ECAPed + artificially aged</td>
<td>16.5</td>
</tr>
</tbody>
</table>

*incomplete breakage of specimen

The absorbed energy of the initial state samples of the alloy is 25.1 J.cm\(^{-2}\). Impact testing of quenched samples was carried out immediately after the solution treatment of initial state and quenching. In this case, the
dissolution of Mg$_2$Si-precipitates takes place during the solution treatment and the precipitation of the strengthening phases (GP–zones, Β′, β′) before impact testing can be ignored. Therefore, the absorbed energy of quenched state is relatively high (54.8 J.cm$^{-2}$).

After ECAP and natural ageing treatment, the absorbed energy decreased markedly reaching 12.5 J.cm$^{-2}$. This value is about 2 times lower than that of the industrially processed (initial state) analyzed alloy. The reason of this toughness loss is first of all the intensive and heterogeneous strain hardening during ECAP. A moderate improvement of the notch toughness was induced by the application of artificial ageing treatment for ECAP-ed state instead of natural ageing. This phenomenon can be outlined by the low recovery of severely deformed alloy microstructure and relaxation of internal stress during artificial ageing, which also improved the strength and elongation during tensile testing. Of course, the coarse irregular multicomponent intermetallic particles, dispersive particles and the assumed precipitates of strengthening phase have also some detrimental effect on the notch toughness of the ECAP-ed and aged EN AW 6082 alloy. The toughness values were raised if sample processing without ECAP was made. Toughness increasing is credit on states without strain hardening. Better toughness value in state after WQ with comparison in slow cooling state is depend on embrittlement effect from precipitation strengthening recognize after slow cooling.

The comparison treatment conditions and obtained mechanical and plastic properties for EN AW 6082 alloy are given in Table 4. from which results:
- the best toughness value was achieved after processing by ($T_{SA}$=550°C/1.5h+WQ) where no strain hardening and precipitation strengthening were occurred
- the lower toughness value was achieved after processing by (400°C/2.5h + $v_{an}$=100°C/h) where no strain hardening but precipitation strengthening were occurred
- the mean toughness values were achieved after processing by (solution annealing + cooling + ECAP + ageing) where strain hardening and precipitation strengthening were observed.

Optimal selection of treatment conditions allows for medium– strength EN AW 6082 alloy to achieve the equivalent properties with high – strength EN AW 7075 alloy.

<table>
<thead>
<tr>
<th>Table 4. The comparison of treatment states, mechanical and plastic properties EN AW 6082 alloy.</th>
</tr>
</thead>
<tbody>
<tr>
<td>State</td>
</tr>
<tr>
<td>Initial state</td>
</tr>
<tr>
<td>$T_{SA}$=400°C/2.5h+$v_{an}$=100°C/h</td>
</tr>
<tr>
<td>$T_{SA}$=400°C/2.5h+$v_{an}$=100°C/h+2x ECAP/20°C</td>
</tr>
<tr>
<td>$T_{SA}$=400°C/2.5h+$v_{an}$=100°C/h+3x ECAP/20°C</td>
</tr>
<tr>
<td>$T_{SA}$=400°C/2.5h+$v_{an}$=100°C/h+4x ECAP/20°C</td>
</tr>
<tr>
<td>$T_{SA}$=550°C/1.5h+WQ</td>
</tr>
<tr>
<td>$T_{SA}$=550°C/1.5h+WQ+3x ECAP/20°C+$T_{SA}$=800h</td>
</tr>
<tr>
<td>$T_{SA}$=550°C/1.5h+WQ+3x ECAP/20°C+$T_{SA}$=100°C/30h</td>
</tr>
<tr>
<td>$T_{SA}$=550°C/1.5h+WQ+3x ECAP/20°C+$T_{SA}$=170°C/1.0h</td>
</tr>
</tbody>
</table>

### 3.4. Microstructure

Recrystallized polyedric microstructure of initial state with grain size about 6 μm is shown in Fig. 9. A presence of irregular intermetallic particles of Al(FeMn)Si phase, undissolved Mg$_2$Si particles and fine Mn-rich dispersive particles were observed in the initial state’s microstructure. The role of Mn-rich dispersoids is to prevent the solid solution grains growth of Al-based alloy. Negligible growth of equiaxed grains of solid solution to 7.3 μm was confirmed after solution annealing and quenching of alloy initial state [14]. No considerable increase in the grain size was prevented by fine dispersed Mn-rich particles, which inhibited recrystallization process [27-30].

The repetitive ECAP process of quenched alloy state induced a significant change of the quenched polyedric microstructure [15] as shown in Fig. 10. Heterogeneous microstructure of the ECAP-ed and aged alloy indicates a non-uniform deformation along the cross-section of the ECAP-ed specimen. No difference was observed between microstructure of the ECAP-ed state after the natural (800 h) or artificial ageing (30h/100°C) using a light microscope.

Substructure evaluation was observed on thin foils. The state ($T_{SA}$=400°C/2.5h/$v_{an}$=100°C/h + 4x ECAP/20°C) is shown in Fig. 11, from which results formation of cell substructure and subgrain creation. When this state is supplemented with annealing at
Ta=300°C/2h recovery and static recrystallization were observed as shown in Fig. 12. Substructure evaluation of state \( T_a=550^\circ C/1.5h + WQ + 3x ECAP/20^\circ C \) is given in Fig. 13, from which results formation of cell substructure and subgrain creation. When this state is supplemented with artificial ageing state \( T_{AA}=100^\circ C/30h \) no recovery was observed as shown in Fig. 14.

Fig. 9. Microstructure of alloy - initial state.

Fig. 10. Microstructure of the ECAPed and naturally aged alloy state (ID – an intensive deformed region, SD – a sligher deformed region).

Fig. 11. State (400°C/2.5h/+SC+4xECAP/20°C).

Fig. 12. State (400°C/2.5h/+SC+4xECAP/20°C +300°C/2h).

Fig. 13. State (550°C/1.5h+WQ+3xECAP/20°C).

Fig. 14. State (550°C/1.5h+WQ+3xECAP/20°C +100°C/30h).
4. Conclusions

Based on experimental results the following conclusions can be made:
- complete dissolution of Mg₂Si particles was observed above the temperature 560°C
- hardness delay above the temperature 560°C was occasioned with changes of Al(FeMn)Si intermetallic particles character
- as desirable solution annealing temperature 550°C was determined
- with increasing of ECAP passes after processing \((T_{SA}=400°C/2,5h/v_a=100°C/h)\) raising of mechanical properties was observed
- when after processing \((T_{SA}=400°C/2,5h/v_a=100°C/h+4x\ ECAP/20°C)\) samples are annealed at various annealing conditions, then hardness decreasing was observed with raising of annealing temperature and time
- when after processing \((T_{SA}=550°C/1,5h+WQ+3x\ ECAP/20°C)\) samples aged at three regimes, then maximal mechanical properties were obtained with artificial ageing condition 100°C/30h and adequate mechanical properties with natural ageing at 20°C/800h
- the best toughness and plastic values and lowest mechanical properties were achieved by sample processing without ECAP application, because minimal strain hardening and precipitation strengthening effects were occurred
- initial state recrystallized polyedric microstructure with grain size of about 6 μm
- diameter of grain size after solution annealing and water quenching showed negligible growth on 7.3 μm
- with application of sophisticated treatment regime \((T_{SA}=550°C/1,5h+WQ+3x\ ECAP/20°C+T_{AA}=100°C/30h)\) is possible with EN AW 6082 alloy to obtain property level specific for EN AW 7075 alloy
- optimal treatment regime generated UFG structure with mean diameter of subgrains on level of 0,26 μm

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References