Increasing strength, ductility and impact toughness of ultrafine-grained 6063 aluminium alloy by combining ECAP and a high-temperature short-time aging

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Abstract. Since fully-dense ultrafine or nanocrystalline bulk materials can be processed, there has been an increasing scientific interest in several plastic deformation (SPD) procedures, particularly in the last decade. Especially the equal-channel angular pressing (ECAP) has widely been investigated due to its ability of producing billets sufficiently large for industrial applications in functional or structural components. The significant strength increase based on grain refinement is typically accompanied by a significant decrease in ductility and toughness. Within this work, a new methodology was applied for combining ECAP with a subsequent high-temperature short-time aging for the 6063 aluminium alloy. An increase in strength, ductility as well as impact toughness regarding its coarse grained counterparts was reached. More precisely, ultimate tensile strength, elongation to failure and impact toughness were increased by 46%, 21% and 40% respectively. This was observed after only one run of ECAP at room temperature in a solid-solution treated condition and an aging at 170°C for 18 minutes. The regular aging time for maximum strength at 170°C is around 6 hours. Longer exposure times lead to recrystallisation and, as for regular aging, it leads to overaging, both causing a decrease of properties. The work demonstrates a strategy for an efficient processing of commercial Al-Mg-Si alloys with outstanding mechanical properties.

1. Introduction
For property optimization of metallic materials by creating an ultra-fine grained microstructure, the ECAP deformation has gained acceptance during the last years [1-3]. During this procedure, a rod-shaped semi-finished product is pressed through an angled channel. When passing the deformation zone (cutting angle of channels), a large shear strain is implemented, which is, compared to extrusion pressing, completely homogenous above the cross-section with a very small fringe.

2. Experimental procedure
The deformation of materials took place in an ECAP prototype tool which is equipped with movable parts on the channel walls to reduce friction. The basic structure is shown in Figure 1. The shear of the used channel angle of $\Phi = 90^\circ$ is $\gamma \sim 2$ which is equivalent to a comparable deformation of $\varphi \sim 1.1$ [4]. Since the tool’s entry and exit channels have the same cross-section, repetitive compressions for cumulating the deformation amount can be attached. In the following text, the number of passes is indicated by the continuous index $N$. 
The majority of current researches deals with unalloyed materials or model alloys which are examined in a way that is irrelevant for practical usage (e.g. soft-annealed or highly-aged states of precipitation-hardened alloys, see [5] as an example). In the present paper, a precipitation-hardened aluminium alloy of technical purity in a completely aged state is accounted for. Table 1 shows the chemical composition of the aluminium alloy used for the ECAP deformation.

Table 1: Chemical composition (wt. %) of the investigated aluminium alloy

| Denomination | Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Zn  | Ti  | Al  |
|--------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| EN AW-6060*  | 0.43| 0.16| <0.01| <0.01| 0.52| <0.01| <0.01| <0.01| balance |
| EN AW-6063   | 0.68| 0.16| 0.01| 0.01| 0.70| 0.002| 0.04| 0.01| balance |

*material for metallographic researches

The characteristic mechanical behaviour in the form of quasistatic technical stress-strain curves, was determined by A3-cylindrical-tension test specimen. Furthermore, the notched-bar impact work was determined by an instrumented notched-bar-impact bending test in compliance with DIN EN ISO 14556 using miniature Charpy V-notch specimen (4 x 4 x 40 mm). The microstructure was valuated using images from an optical and electron microscope of the low-alloyed aluminium EN AW-6060. The technical basis formed a field-emission scanning electron microscope (Neon40) from Zeiss.

3. Results and discussion

3.1. Development of Strength and Ductility

For the researches, material EN AW-6063 was solution heat-treated at 530°C for one hour (index -W), and then deformed up to six times by ECAP route E (see [3]).

The development of the mechanical values yield point $R_{p0.2}$, tensile strength $R_{m}$, uniform elongation $A_g$, and elongation to failure $A_3$ in Figure 2 show a strong increase of strength values with a high loss
of ductility. The properties of the maximum-aged heat-treatment state (index –T6) of the undeformed alloy are used as reference values. After six deformations, yield point and tensile strength of the ECAP aluminium are 84% and 74% above the T6 reference state. However, uniform elongation, which is important for constructive purposes, drops to 42% of the undeformed state.

This lost ductility shall be regained by a downstream heat treatment at 170° C. The consequences of the downstream heat treatment on the simple-shaped material are shown at the right side of Figure 2. Annealing for 18 minutes at a temperature of 170°C leads to a retrieval of ductility whereas the uniform elongation increases from 3.5% to respectable 7%, which almost matches the value of the reference state T6 (8%). Strength does not change. In accordance with the study on AA6060 (presented in ref. [6]) the maximum ductility achieved during aging decreases also for the AA6063 with increasing number of passes and/or decreasing aging temperature, while the maximum strength behaves vice versa. Since the billets are designated for safety relevant applications the focus was to optimize the damage tolerance and thus only one pass is sufficient.

Compared to the reference T6, this optimized state shows an increase in tensile strength of 46% as well as in elongation to failure of 21%.

### 3.2. Development of Charpy Toughness

![Force-displacement curves received from Charpy impact tests of simple ECAP shaped EN AW-6063-W, N = 1 with post ECAP heat-treated condition 170°C, 18 minutes compared to the reference EN AW-6063-T6](image)

The notched-bar impact value (NBIV), which results from integrating the load-deflection curve of the instrumental notched-bar-impact bending tests as shown in Figure 3, features an increase of absorbed energy of 40% (compared to the reference state T6) which is due to ECAP deformation and the downstream heat treatment.

**Figure 3.** Force-displacement curves received from Charpy impact tests of simple ECAP shaped EN AW-6063-W, N = 1 with post ECAP heat-treated condition 170°C, 18 minutes compared to the reference EN AW-6063-T6

### 3.3. Microstructure

The intense decrease of grain size due to the implemented shear strain explains the significant increase in strength due to ECAP (see left side of Figure 2).

![EN AW-6060 in normal granular state through an optical microscope (left side), STEM micrograph of ultrafine grained state after N = 8 deformations (right side)](image)

**Figure 4.** EN AW-6060 in normal granular state through an optical microscope (left side), STEM micrograph of ultrafine grained state after N = 8 deformations (right side)

The initial microstructure in Figure 4 (left side) shows an average grain size of 120 µm, which decreased to 310 nm (Figure 4; right side) by an eightfold ECAP deformation. Hereafter, the optimizing heat treatment after the first ECAP deformation shall be discussed. The blurry dark areas in the once-only ECAP shaped state (a) shown in the EBSD image in Figure 5 (left side) indicate strong inner tensions. These dark areas represent the newly formed dislocations which accumulate at the
grain boundaries and inside the grains, and increase the strength of the material. Light areas indicate no tensions inside the grain. The more the aluminium strengthens due to dislocation accumulations the blurrier is the black-white contrast. With the optimizing heat treatment (b), tensions are partially reduced, and the picture contrast is increased. The dislocations can shift, and as a result, the developing small-angle grain boundaries contribute the grain refinement. This microstructure recovery allows a retrieval of ductility.

The high-resolution TEM image of the once ecaped state (a) in Figure 5 (right side) clearly shows the dislocation accumulations (white arrows) which partially form small-angle grain boundaries. These boundaries match the blurry lines running through the grain in the EBSD image in Figure 5 (left side). After the heat treatment, the TEM image (b) shows small-angle grain boundaries which developed due to the shifting of the dislocations (white arrows). Furthermore, there are finest precipitations close to the free dislocations, acting as nuclei, which preserve the strength of the material during heat treatment.

**Figure 5.** EBSD quality (band contrast) maps (left side) representing the microstructure after N = 1; TEM bright-field images (right side) showing the microstructure after N = 1 for (a) As-processed condition without subsequent aging and (b) high-temperature aged at 170 °C for 18 minutes [6]

### 4. Summary and conclusion

The presented results demonstrate the high potential of the combined process that induces high plastic strain like ECAP for EN AW-6063 with an appropriate aging treatment for achieving high strength, high charpy impact toughness and high ductility, compared to the commercial coarse grained counterparts. The effects occur after a single pressing and a short high-temperature aging, making the process attractive for practical applications.

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