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Structure Development after Twist Channel Angular Pressing

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The twist channel angular pressing technology, based on the conventional equal channel angular pressing supplemented with an additional twist, was developed to increase the efficiency of severe plastic deformation imposed into a processed material during a single pass. The study investigated and compared sizes and preferential orientations of grains within commercial purity Al processed by TCAP and ECAP. The results revealed the grain refinement process to be very effective during TCAP (almost 90% of grains were smaller than 5 μm after a single pass). Transmission electron microscopy was used to study sub-structure development within a TCAP-processed sample, the results showed grains with locations featuring accumulated dislocations and highly developed sub-grains after a single TCAP pass. The occurring softening processes also contributed to the fact that the TCAP-processed structure featured no strongly prevailing preferential grains orientation. The study was supplemented with a brief analysis of mechanical properties of TCAP and ECAP processed samples, which showed very favourable results for the TCAP-processed one.

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1. Introduction

The everlasting hunger for new materials and continuous enhancement of the properties of the well-established ones has led to the development of innovative forming methods, such as the methods of severe plastic deformation (SPD) [1]. The SPD methods can be characterized as continuous (featuring the capability to process larger billets), e.g. equal channel angular pressing-conform (ECAP-Conform) [2, 3], continuous confined strip shearing [4], constrained groove pressing [5], etc.; and discontinuous (primarily featuring great versatility), such as high pressure torsion [6, 7], twist extrusion [8], torsion extrusion [9], (variable lead) axisymmetric forward spiral extrusion [10, 11], cyclic extrusion compression and cyclic expansion extrusion [12, 13], and, last but not least, ECAP [14, 15], and thereto related technologies — non-ECAP (NECAP) [16], ECAP with partial back pressure (ECAP-PBP) [17], ECAP with back pressure (ECAP-BP) [18, 19], twist channel multi AP (TCMAP) [20], and twist channel AP (TCAP) [21].

TCAP was relatively recently developed by Kocich et al.; the conventional ECAP die is there supplemented with an additional twist (described in detail e.g. in Refs. [22, 23]) providing the die with the ability to impose an increased amount of strain into the processed billet during a single pass. The previously performed experiments and numerical predictions proved that TCAP increases substantially the strain imposed during a single pass and its homogeneity compared to ECAP [21, 22, 24]. However, detailed studies of the influence of TCAP on the development of substructure are scarce [23, 24].

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The aim of this work is to characterize grain sizes and orientations together with substructure development within a commercial purity (CP) aluminium billet processed by TCAP. The results are compared with another sample processed by conventional ECAP under identical conditions. The analyses comprising evaluation of grain sizes and orientations are supplemented with the results of tensile testing. Last but not least, samples of both the processed materials were subjected to TEM investigations of substructure development.

2. Experimental

Primarily due to its low flow stress and very high stacking fault energy (SFE) [25], the experiments were performed with CP Al (99.7%). The to be extruded $12 \times 12 \times 120$ mm^3 billets were annealed at 400 °C for 1 h and processed by ECAP and TCAP at room temperature. The processing rate was 3 mm/s and MoS_2 was used as the lubricant.

The grain sizes and orientations were analysed via SEM on samples' longitudinal cuts in the locations of their processing axes. Longitudinal cuts were selected in order to better demonstrate the development of low and high angle boundary grains (LAGBs and HAGBs, subgrains and full grains). All the samples for scanning electron microscopy (SEM), electron backscattering diffraction (EBSD) analyses were ground on SiC papers and polished electrolytically. The observations were carried out with the 0.5–1.0 μm scan step using a Tescan Lyra 3 FIB/SEM microscope with a NordlysNano EBSD detector. Grains orientations evaluations were carried out having 15° as the LAGBs and HAGBs misorientation limit. The TEM foils were prepared with the help of a twin-jet electro-polishing machine from samples' axial longitudinal cuts and examined using a JEOL 2100F TEM. The tensile tests were performed with specimens taken from axial areas of the extruded samples using an Instron

3382 machine. For the tests, the cross-head velocity was 0.5 mm/min and the strain rate was 0.56×10^{-3}/s.

3. Results and discussion

3.1. Grain size

The undeformed Al had coarse grains, almost 70% of the grains were smaller than 50 μm and their average diameter was less than 40 μm (Fig. 1a). After ECAP, the grains got refined to the average diameter of approximately 7 μm (Fig. 1b). The results are comparable to those reported by others, for example Cabibbo et al. [26] reported almost 10 times grain refinement after a single ECAP pass of CP Al at room temperature, and so did El-Danaf [27].

The substantially higher strain imposed by TCAP caused notable decrease in the average grain diameter; the majority of the grains (almost 90%) in the investigated area of the TCAP-ed structure was smaller than 5 μm and the average grain diameter decreased to slightly more than 2 μm (Fig. 1c). A single TCAP pass thus exhibited higher efficiency of grain refinement than single pass ECAP. The grain refinement results were also better than results for two-pass ECAP reported elsewhere [27, 28]. This favourable effect can be attributed to the character of the TCAP process, which basically combines two SPD methods, ECAP and twist extrusion, in a single die [21, 22, 29]. Firstly, the twist section of the TCAP die imposes the shear strain primarily to the peripheral regions of the processed sample. Subsequently, the central region of the sample is subjected to shear strain in the bending part of the die. The TCAP die as a whole thus provides new severe deformation metal forming possibilities.

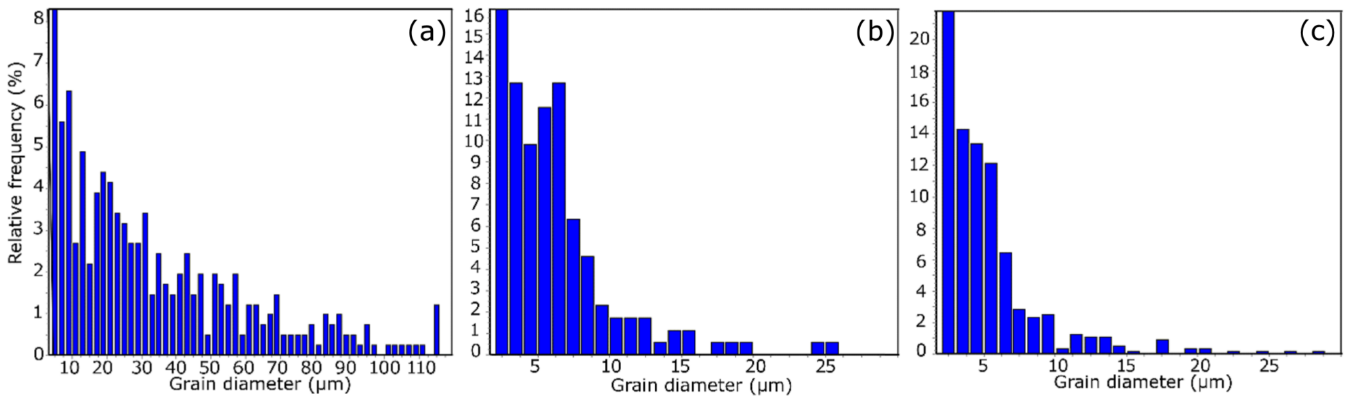

Fig. 1. Grain sizes distribution for the original material (a), after ECAP (b), and after TCAP (c).

3.2. Grains orientations

As demonstrated by Fig. 2a, the undeformed Al featured a relaxed structure with more or less random grains orientations. After ECAP, the grains slightly elongated in the main direction of the effecting shear strain and started to gain the preferential ⟨111⟩ orientation (Fig. 2b). The original grains got fragmented to smaller grains and subgrains more or less retaining the orientations of the original structural units, however, slight deviations of the new (sub)grains within the original grains commence to be notable. The observed facts corresponded e.g. to the results of studies by El-Danaf et al. [27, 30], who reported notable increases in the volume fractions of grains misorientations between 5 and 15° (around the limit between LAGBs and HAGBs) after a single pass ECAP. This is also in accordance to the observed (sub)structure development, as discussed further in Sect. 3.3.

The relatively high imposed strain during a single TCAP pass [23] caused the original grains to fragment into subgrains (and subsequently form new grains, as will be seen better in Sect. 3.3), which formed clusters with identical/similar crystallographic orientations. However, the volume fractions of the structural units rotated to the preferential directions of ⟨001⟩, ⟨011⟩ and ⟨111⟩ were similar and thus eventually no preferential orientation prevailed (Fig. 2c). Moreover, more than one third of the grains were rotated up to 45° between two of the main orientation directions. Therefore, TCAP imposes higher strain than ECAP within a single pass, however, due to strain path changes [24], the grains orientations are more random than after ECAP.

3.3. Substructure development

Substructures of the samples processed by ECAP and TCAP are depicted in Fig. 3a and b, respectively. The ECAP-ed sample featured slightly elongated original grains with newly emerged dislocations arranging into dislocations walls and cells, which is the pre-stage of formation of subgrains introduced by the imposed strain [31]. The TCAP-ed sample also featured the original grains with dislocations-formed substructure, however, the sample already exhibited new grains and highly developed subgrains starting to transform into high-angle boundary grains. The sizes of the subgrains introduced by TCAP were comparable or smaller than the smallest grain size achievable by ECAP [32], which points to the promising TCAP structure refining potential, especially when multiple passes would be used.

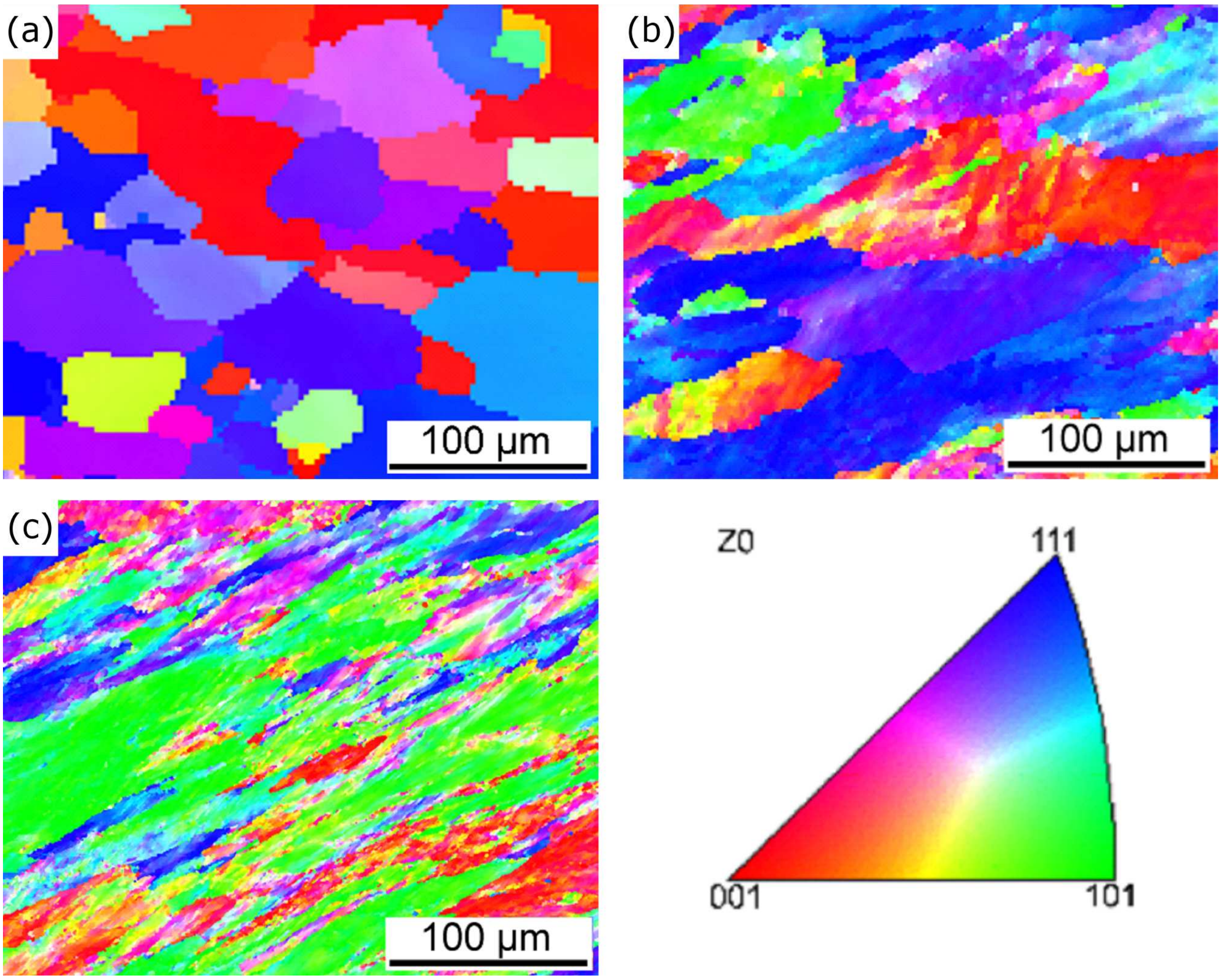

Fig. 3. Substructures after ECAP (a) and TCAP (b).

The structure also exhibited several grains undergoing recovery (evident grain boundary movement). The high pressure and high imposed strain lowered the activation energy for recovery-recrystallization start, which resulted in dynamic recovery occurring during/right after processing [33]. Another structural feature observed especially for the TCAP-ed material was the presence of secondary particles, most of which had the diameters of around 10 nm, however, larger particles could also be seen. A more detailed study of the precipitated particles was published elsewhere [23]. Although the larger particles do not support the pinning effect as notably as the smaller ones do, moving dislocations can (repeatedly) fragment them to smaller pieces, which can significantly contribute to the strengthening effect [34].

3.4. Mechanical properties

The stress–strain curves for the ECAP-ed and TCAP-ed materials, together with a schematic depiction of the shape and location of samples cutting from the extruded billets, are summarized in Fig. 4. They are compared with the stress–strain curve for the undeformed material, which exhibited the lowest strength ($\approx$ 120 MPa) and the highest plastic deformation before failure. The strength increased to more than 250 MPa after ECAP. Strengthening of Al approximately by the factor of two after a single pass ECAP was also reported e.g. by Xu et al. [35].

Nevertheless, even more significant strengthening was observed for the sample processed via TCAP, the ultimate tensile strength of which exceeded 330 MPa. Such a notable strengthening can be attributed to the substantial grain refinement (as shown in Sect. 3.1) and the presence of fine secondary particles (Sect. 3.3), which created barriers for pinning of dislocations and subgrains boundaries [36, 37]. Despite the fact that the volume fraction of the fine precipitates in the TCAP-ed structure was relatively small and the strengthening can therefore primarily be attributed to the grain refinement, Humphreys et al. [33] reported the effect of precipitates on pinning of boundaries and therefore on the final strengthening to be substantial even for their smaller amounts.

The serrations occurring especially at the endings of the stress–strain curves were primarily caused by the presence of the precipitated particles together with

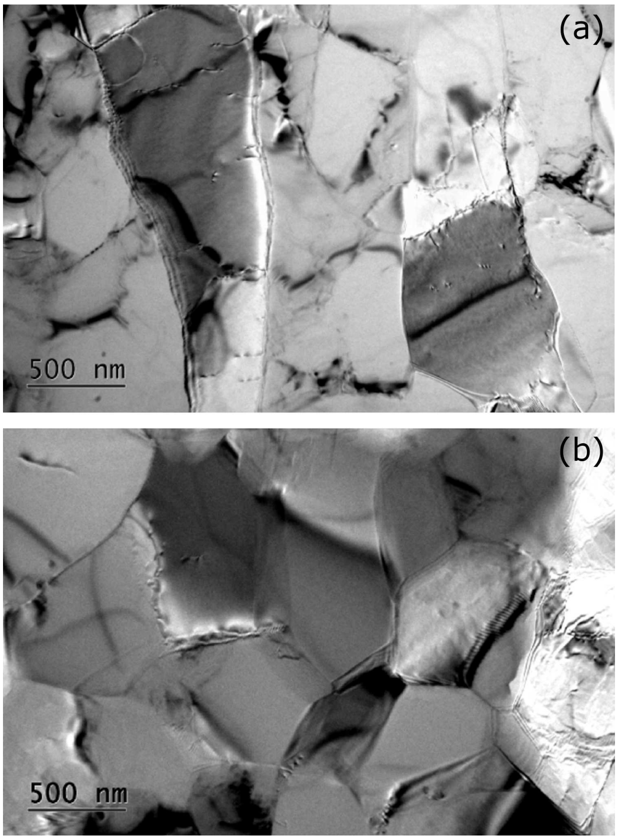

Fig. 4. Stress-strain curves for undeformed, ECAP-ed and TCAP-ed materials.

the restricted dislocations movement. Such serrations are typical for Al-based alloys exhibiting the Portevin–LeChatelier (PLC) effect, which is typically provoked by dynamic strain ageing, during which atoms of the solid solution diffuse to moving dislocations and thus restrict their movement [38, 39]. The processed material exhibited precipitated particles and quite a substantial number of dislocations, however, the presence of other atoms dissolved in the solid solution can also be suspected, because virtually no commercial Al-based alloy is formed of 100% Al [33]. Moreover, the occurring dynamic strain ageing results in further precipitation and thus introduction of further obstacles for dislocations movement. Therefore, the contributions of precipitated particles and solute atoms on the overall strengthening can only hardly be determined.

4. Conclusion

The focus of this study was on demonstration of the efficiency of the recently developed SPD process, twist channel angular pressing — TCAP. The analyses of the TCAP-processed material showed significant grain refinement and substantial substructure development. The TCAP-processed structure exhibited the average grain diameter of slightly more than 2 μm, which was substantial grain refinement when compared to the original structure, and also to the structure processed by ECAP, which exhibited the average grain diameter of around 7 μm. The investigations of TCAP-ed substructure revealed the degree of its development to be higher than achievable by ECAP. The structural changes imparted by TCAP resulted in notable increase in strength of the processed material, from original 120 MPa to more than 330 MPa. The strengthening was caused primarily by the grain refinement and increase in dislocations density, but also by the presence of fine precipitated particles restricting dislocations movement. Despite the fact that this study was performed on CP Al and the concrete behaviour of individual materials will slightly differ, the occurring phenomena and thus the effect of the TCAP on any material can be supposed to be very similar.

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