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# Sub-structure and mechanical properties of twist channel angular pressed aluminium

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ABSTRACT

The twist channel angular pressing (TCAP) method was developed with the aim to increase the effectiveness of severe plastic deformation (SPD) technologies. This study was focused on investigations of grains sizes, textures and sub-structure within a TCAP-processed commercial aluminium sample. Measurements of microhardness and tensile strengths were performed to evaluate mechanical properties of the processed material. To point out the effectiveness of TCAP, the results were compared to Al samples processed by equal channel angular pressing (ECAP) via A and Bc routes. Almost 70% of the grains within the sample processed by a single TCAP pass were smaller than 5 μm comparing to approximately 40% and 50% within samples processed by two ECAP passes via A and Bc routes, respectively. The texture of the TCAP sample exhibited a combination of A ideal and B fibre preferential texture components and its sub-structure exhibited a higher degree of development than the sub-structures in all the ECAP-processed samples. The TCAP sample also exhibited the highest increases in the mechanical properties. The microhardness was more than twice as high as for the original state, while the yield strength achieved almost 230 MPa.

## 1. Introduction

The most significant advantage of severe plastic deformation (SPD) technologies is their ability to effectively refine structural elements via imposing high amounts of shear strain consequently resulting in enhancement of physical and mechanical properties of the processed materials [1,2]. Materials processed by SPD technologies can find their use in various high-strength industrial applications [3], as well as in implant industry [4].

One of the mostly investigated SPD technologies is the equal channel angular pressing (ECAP) method. A material processed by ECAP is deformed primarily by simple shear and the amount of the imposed strain depends on various processing parameters including material characteristics, processing temperature, friction and geometry of the die [5–7]. On the other hand, homogeneity of strain distribution across the cross-section of a sample after one ECAP pass can be relatively non-uniform [8]. Simple shear is the main deformation mechanism also for another SPD process, the twist extrusion (TE) method [9], for which the inhomogeneity of strain throughout the cross-section of a processed sample is obvious [10].

Considering the ECAP-based methods, higher imposed strains usually contribute to structure refinement. However, a very high imposed strain can lead to a saturation of structure related with achievement of an equilibrium state between generation and annihilation of dislocations, which significantly reduces the possibility of further structure refinement [11]. Although an increasing imposed strain generally leads to a greater structure refinement, it can also lead to an increased inhomogeneity of strain distribution under certain conditions (e.g. unfavourable influence of die geometry, selected deformation route, friction conditions, temperature, extrusion velocity etc).

The twist channel angular pressing (TCAP) method, recently invented in accordance to the contemporary trend of increasing the effectiveness of SPD processes, basically consists of the combination of the ECAP and TE processes (Fig. 1).

Previously performed experiments and numerical predictions for TCAP showed a substantially higher imposed strain when compared to ECAP [12]. During the ECAP process, the one and only deformation zone is situated into the channel bending, (defined by the $\varphi$ and $\psi$ angles), while during the TCAP process, the sample extruded through the channel is among this deformation zone affected also by the twist deformation zone (defined by $\beta$ a $\omega$ angles). Therefore, the total strain which can possibly be imposed into a sample during a single deformation pass is higher for the TCAP process (~2.3 for $\varphi = 90°$ and $\omega = 90°$) than for the ECAP process (~1.15 for $\varphi = 90°$). Moreover, they showed a favourable influence of this method on homogenization of strain throughout the cross-section of a deformed sample [13,14]. The possibility to predict homogeneity of strain and to calculate with possibly variable deformation parameters (temperature, extrusion velocity etc.) are the primary advantages of numerical simulations over

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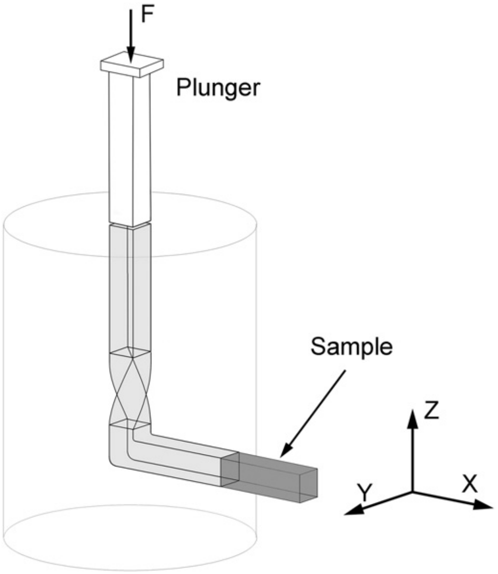

Fig. 1. Schematic diagram of the TCAP die with indications of sample axes.

mathematical calculations, such as the calculation according the Iwahashi's equation [15]. One of the previous studies also dealt in detail with the influence of die parameters, such as channel bending angle and twist slope angle, on the imposed strain [16]. However, none of the studies carried out so far was focused on texture investigations and sub-structure characterization via scanning and transmission electron microscopies (SEM, TEM).

The aim of this paper was to characterize sub-structure, grain size and texture within a sample processed by a single TCAP pass and to relate it to mechanical properties, microhardness and tensile strength in particular. For an on-site comparison and to increase reliability of the study, two samples were processed by two ECAP passes, A and Bc routes, and subsequently analysed. The basic differences between all the processed samples were then pointed out.

## 2. Experimental

### 2.1. Material

The experiments were performed using commercial Al (99.17%), which was selected for its low flow stress and a very high value of stacking fault energy (SFE) determining the intrinsic material properties generally favourable for demonstration of structural changes induced by severe plastic deformation [17]. Billets with the 12x12x120 mm$^3$ dimensions were at first annealed at 400 °C for 1 h. The extrusions were performed at room temperature using a NMT300 ECAP-technology hydraulic press either by one ECAP pass, two ECAP passes (routes A, Bc) or one TCAP pass. The billets were extruded at the rate of 3 mm s$^{-1}$ using MoS$_2$ lubricant. Samples for subsequent analyses were cut using a CNC electro-erosive wire cutting machine.

The grain sizes were analysed on YZ cross-sectional cuts perpendicular to the extrusion axes of the samples (axis X in Fig. 1), while texture observations were performed on XZ longitudinal cuts parallel to the extrusion axis of the bottom outlet part of the ECAP (TCAP) channel. The locations of the analyses were for both cases selected to be in the middle distance between the central axis and upper surface of the sample. The samples for SEM investigations were ground on SiC papers, pre-polished using 0.3 μm alumina compounds and finally polished electrolytically. The observations were performed using a Tescan Lyra 3 FIB/SEM microscope equipped with a NordlysNano EBSD (electron backscattered diffraction) detector. EBSD analyses were performed on samples tilted by 70° with the scan step of 0.5–1.0 μm and an accelerating voltage of 20 kV. Evaluations were performed considering the 15° misorientation as the limit between low-angle and high-angle grain boundaries and the maximum 20° deviation from the ideal texture positions. The 3D scan was acquired using FIB milling and EBSD data acquisition, the configuration of which was in accordance to the static set-up introduced in [18]. Before milling, which was conducted at 30 KeV, the surface of the examined block was covered with a protective Pt layer. The beam current was 800 pA, while the voxel size for 3D EBSD maps was 0.2 μm. The foils for TEM were prepared by a twin-jet electro-polishing device and examined using a Jeol 2100F TEM device operating at 200 kV.

Mechanical properties were analysed via tensile testing and Vickers microhardness (HV100) measurements. Tensile tests were performed on specimens taken from central regions of the extruded samples using an Instron 3382 machine with cross-head velocity of 0.5 mm/min (strain rate of $0.56 \times 10^{-3}$ s$^{-1}$). The microhardness was measured across cross-sections of all the samples with the load of 100 N and the indentation time of 15 s using a FM ARS 900 (Future Tech) microhardness testing machine equipped with a diamond indenter.

## 3. Results and discussions

### 3.1. Grain sizes

The grain size analyses showed the unprocessed Al to have the coarsest grains. For this sample, several individual grains had the diameter of almost 150 μm, although 69% of the grains were smaller than 50 μm and the average grain diameter was ~39 μm (Fig. 2a). The grains were significantly refined after one ECAP pass having the average grain diameter slightly above 10 μm. Although large grains were still present, the majority of grains were smaller than 50 μm and >90% of the grains were smaller than 20 μm (not shown here). Subsequent extrusions of the billets via second passes resulted in further grain refinements. The average grain size after two A route passes was ~8 μm, while after two Bc route passes (route Bc) it was ~7.4 μm. The content of the grains smaller than 5 μm was 40% for the A route sample (Fig. 2b) and 50% for the sample processed by Bc route (Fig. 2c). The results showed that the most significant relative grain refining effect was achieved during the first pass, even though grains were refined also during both the second passes. Cabibbo et al. [19] reported similar results for CP Al, for which they achieved grain refinement by 10 times after one pass ECAP at room temperature, while the grain size further decreased only approximately by half during each subsequent pass. This phenomenon is given by dislocations rearrangement mechanisms [20]. Models describing the generation, movement, locking and annihilation of dislocations induced by severe plastic deformation were already introduced [21], including the one by Hosseini and Kazeminezhad [22] suitable especially for FCC metals with high SFE.

Comparison of TCAP with both the two ECAP passes indicated very high grain refinement effectiveness of TCAP. The average grain diameter within the sample processed by one pass TCAP was <6 μm, which was smaller than for all the ECAP-processed samples. Moreover, the high imposed strain caused nearly 70% of the grains in the examined central area of the sample cross-section to be smaller than 5 μm (Fig. 2d). Regarding the ECAP deformation routes, Dobatkin et al. [23] have already proven the Bc route to influence the most effectively the grain refinement and the consequent increase in strength properties. This will be confirmed in Section 3.7.2 of this paper. However, one pass TCAP exhibited even higher efficiency of grain refinement, which can be attributed to the fact that this method combines ECAP with TE and therefore it can be considered as a combination of Bc and A ECAP deformation routes within a single pass. At the first TCAP deformation stage, especially the peripheral regions of the sample experienced substantial shear imposed by the twist section of the die, while the strain subsequently experienced by the material in the bending part of the channel is imposed primarily to its central region [10,12,16]. TCAP thus introduces new possibilities of mutations of the existing ECAP deformation routes,