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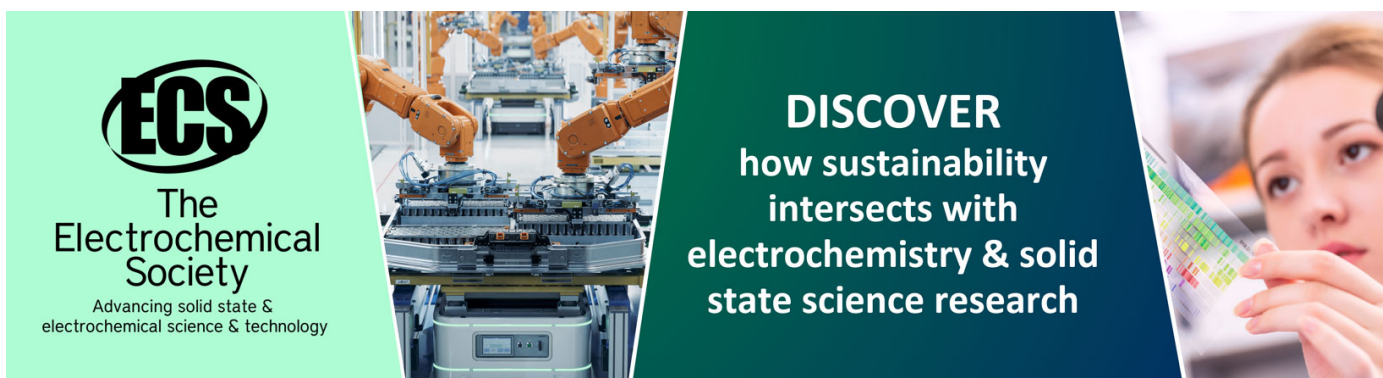
## Comparison of mechanical and microstructural properties of conventional and severe plastic deformation processes

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# Comparison of mechanical and microstructural properties of conventional and severe plastic deformation processes

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**Abstract.** The effect of the deformation processes on yield stress, Vickers microhardness and dislocation density were investigated using commercial purity (A1050) and alloyed aluminum (Al 6082). For the evolution of the dislocation density X-ray line profile analysis was used. In the large plastic strain range the variation of mechanical and microstructure evolution of A1050 and of Al 6082 processed by equal channel angular pressing are investigated using route B<sub>C</sub> and route C. In the plastic strain range up to 3 plane strain compression test was used to evaluate mechanical properties. The hardness and the yield stress showed a sharp increase after the first pass. In the case of A1050 it was found that the two examined routes has not resulted difference in the flow stress. In the case of Al 6082 the effect of the routes on the yield stress is significant. The present results showed that in the comparable plastic strain range higher yield stress values can be achieved by plane strain compression test than by ECAP.

## 1. Introduction

The microstructure of bulk metals can be significantly changed by severe plastic deformation (SPD) processes. The SPD techniques result relevant grain refinement so that the grains are reduced to submicron range, offering a great potential in improving strength. The most popular and most developed SPD technique is the Equal Channel Angular Pressing (ECAP). The billet is pressed through a die consisting of two channels with equal cross-section. Because of the identical cross-sections of the channels, the dimensions of the billet remain unchanged it allows the repetitive pressings to achieve large plastic strain. The intersection of the two channels has an angle usually in the range of 90° to 157° [1]. The deformation occurs to the intersecting plane of the channels. In theory the shear deformation is concentrated in a narrow band, however experiments showed that in practice the shear spread over a deformation zone [2]. During ECAP process the shearing characteristic may be changed by rotating the sample around its axis between each pass. Thus, the different routes have effect on the evaluated microstructure [3]. Deformation by ECAP to large plastic strain resulting the evolution of high-angle boundaries from subgrain boundaries [4][5].

It was shown that the degree of non-monotonicity (DNM) has an effect of the evolved microstructure. The higher value of DNM the greater grain refinement can be achieved [6]. In conventional metal forming processes the evolution of high-angle boundaries cannot be achieved because of the limit of the imposed plastic strain. Usually ECAP and conventional metal forming processes are characterized by non-monotonic and monotonic processes, respectively [7].

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The plastic behaviour of materials depends on the deformation history not only on the current state of deformation. The plastic response of materials in the subsequent deformation can be different from that of the previous stage. The parameter for the change of strain path, or strain path change (SPC), was introduced by Schmitt et al. [8]. In this parameter ( $\theta$ ) the directionalities of the strain or strain rate tensors are considered in the previous and subsequent deformation state.

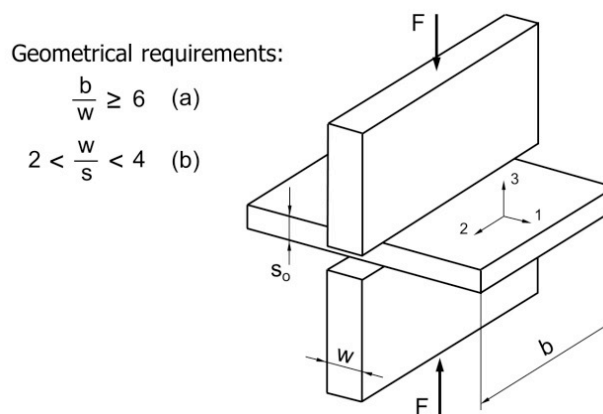
The present work focuses on the difference in flow behaviour and in hardness of samples produced by plane strain compression test (monotonic deformation) and by equal channel angular pressing (non-monotonic deformation). Experiments were conducted on commercial purity (A1050) and on alloyed aluminum (Al6082).

## 2. Experimental procedures

The experiments were performed on A1050 aluminium. Cylindrical and sheet-like specimens were manufactured from the rod-like raw material. The annealing of specimens happened at 370°C through 2.5 hours followed by air cooling. Plane strain compression test was used to determine the material properties under monotonic deformation in the 0-3 plastic strain range. The strain rate was about 0.53 1/s. In the large strain range equal channel angular pressing was investigated. On all deformed specimens Vickers microhardness were measured by Buehler hardness tester at load 200 g for 10 s and the reported value is the average of minimum 5 readings. All of the experiments were conducted at room temperature. The comparison of mechanical properties was done also on Al6082 alloyed aluminium, based on the previous work of Á. Fodor [9]. However, additional measurements were made on the same material at the same annealed state.

### 2.1. Plane strain compression test

The plane strain compression test (otherwise known as Watts-Ford test) was performed on MTS 810 material testing machine. The Watts-Ford method lead to reliable results if the plane strain conditions are kept during the simulation [10]. The schematic drawing of the Watts-Ford test and the geometrical requirements are shown in figure 1.



**Figure 1.** Schematic illustration of the Watts-Ford test and the corresponding geometrical requirements

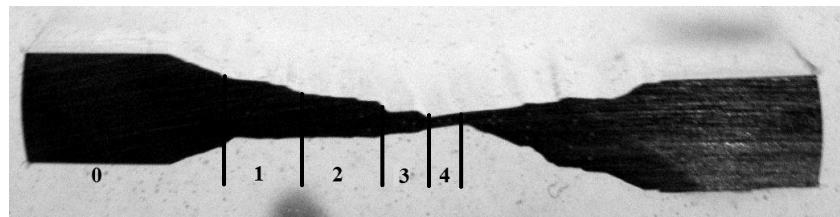
The specimen geometry was 30 mm in width, 3 mm in thickness and 20 mm in length. Four changeable tools were used with the following width: 6.06 mm, 4.40 mm, 2.24 mm and 1.18 mm. With each pair of tools the upsetting was made step-by-step with small deformations. To minimize the effect of friction the specimen were relubricated at each upsetting steps. The flow stress and the equivalent strain obtained as:

$$k_f = \frac{\sqrt{3}}{2} \frac{F}{wb}, \quad \bar{\varepsilon} = \frac{2}{\sqrt{3}} \frac{s_0}{s} \quad (1)$$

where  $w$ ,  $b$ ,  $s$ , and  $s_0$  are the width, the length, the thickness and the initial thickness of the specimen, respectively. On the results of the measurements a Voce type relationship were fitted:

$$k_f = \sigma = \sigma_0 + \sigma_1 \left\{ 1 - \exp \left[ - \left( \frac{\bar{\varepsilon}}{\bar{\varepsilon}_c} \right)^n \right] \right\} \quad (2)$$

where  $\sigma_0$ ,  $\sigma_1$ ,  $\bar{\varepsilon}_c$  and  $n$  are the fitting parameters. Physically, the  $\sigma_0$  is the yield stress and  $\bar{\varepsilon}_c$  is the equivalent strain value, where  $\sigma$  reaches a certain percentage of the saturation stress which given by  $\sigma = \sigma_0 + \sigma_1$ . On each deformation zone Vickers microhardness and X-ray line profile analysis were examined. The cross-section of the specimen deformed by Watts- Ford test is shown in figure 2.



**Figure 2.** Cross-section of the deformed Watts-Ford specimen

## 2.2. Equal Channel Angular Pressing

The ECAP experiments were conducted on hydraulic machine at speed of 60 mm/min. The specimen geometry was cylindrical shaped with 15 mm in diameter and 180 mm in length. Two routes were adopted up to a total number of 16 passes (1, 2, 4, 8 and 16 passes) for A1050 and a total number of 8 passes for Al6082 with dies having  $\phi=90^\circ$  and  $\Psi=10^\circ$ . The samples were lubricated with using graphite based lubricant between each passes. The relation for the equivalent plastic strain and for the average strain rate [11]:

$$\begin{aligned} \bar{\varepsilon} &= \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + \psi \cos ec \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right] \\ \dot{\bar{\varepsilon}} &= \frac{1}{\sqrt{3}} \left[ 2 \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + \psi \cos ec \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right] \frac{\sqrt{2}v}{\psi w} \end{aligned} \quad (3)$$

where  $N$  is the number of passes,  $\phi$  is the intersecting angle of the two channel,  $\Psi$  defines the arc of curvature at the outer point of intersection of the two channels,  $v$  is the ram speed and  $w$  is the diameter of the billet. Based on equation (3) the calculated average strain rate is 0.53 1/s. The Vickers microhardness measurements were performed on each pressed samples normal to the extrusion direction. For X-ray diffraction measurements, from the first and the last ECAPed specimens, cylindrical samples were manufactured. To determine the mechanical properties of the workpieces proportional tensile test specimens were machined parallel to the extrusion direction. The tensile tests were performed on Instron 8501 testing machine with crosshead velocity of 1 mm/min. The strain path

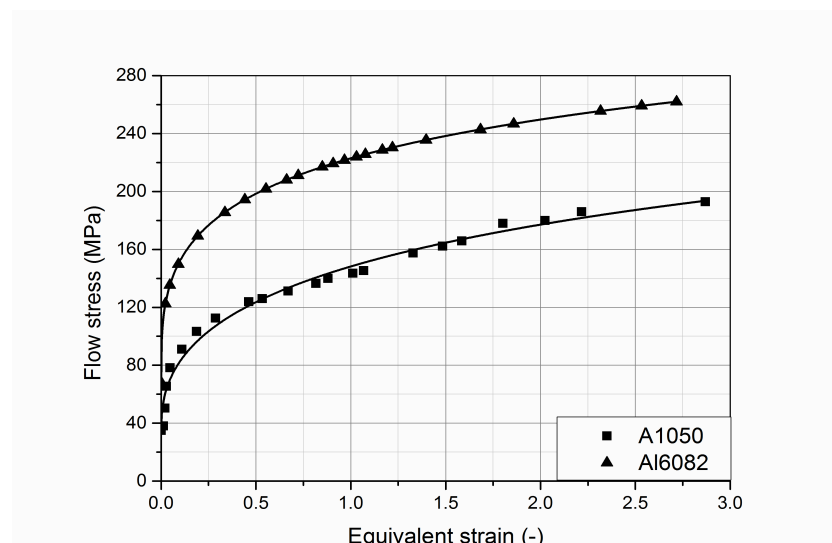
change parameter for ECAP with a  $90^\circ$  die followed by uniaxial tension in the extrusion direction has been reported to be  $\theta = \sqrt{3}/2$  by Li [12], which is consistent with the quasi-monotonic strain path. There is no significant change in the magnitude of the change of strain path. It is assumed that tensile test, if the specimen is machined parallel to the extrusion direction, is a suitable test to determine the stress state of ECAPed specimen.

### 3. Results and discussion

The results of the Watts-Ford measurements and the fitted experimental functions are shown in figure 3. The yield stress of A1050 and of Al6082 was 35 MPa and 68 MPa, respectively. The parameters of the fitted function are summarized in table 1. It can be seen that the flow stress increased to about 195 MPa for A1050 and about 262 MPa for Al6082. It is assumed, in cold working condition, the flow curve increases monotonically with equivalent plastic strain and tends to a saturation value.

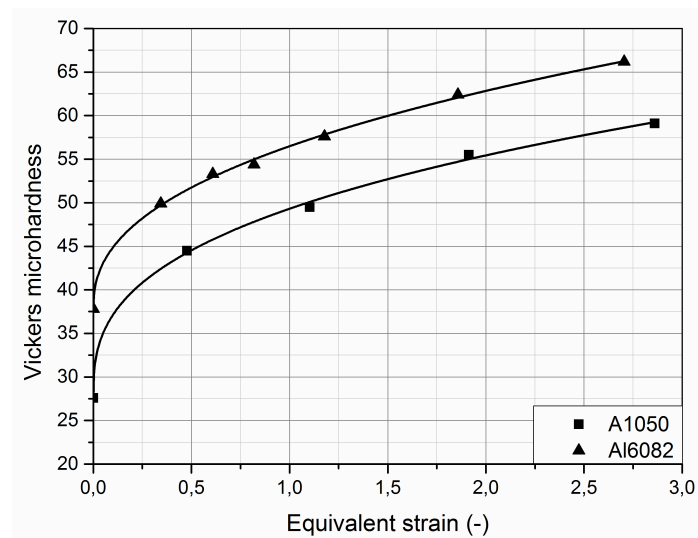
**Table 1.** The parameters of equation (3) for A1050 and for Al6082 obtained by Watts-Ford test.

| Material | $\sigma_0$ (MPa) | $\sigma_1$ (MPa) | $\bar{\varepsilon}_c$ (-) | n(-)  |
|----------|------------------|------------------|---------------------------|-------|
| A1050    | 35               | 302.7            | 5.69                      | 0.436 |
| Al6082   | 68               | 339.3            | 4.48                      | 0.33  |



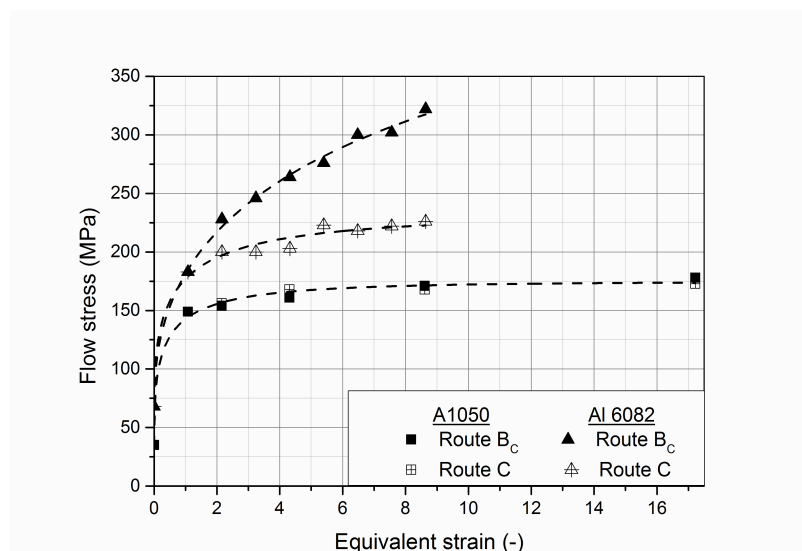
**Figure 3.** Results for A1050 and Al6082 obtained by Watts-Ford test

The initial Vickers microhardness of the annealed A1050 material was 27.6 and after the last compression step increased up to 59. In the case of the alloyed aluminum the initial value was 37.8 and it almost doubled at the plastic strain value of 2.7. The Vickers microhardness values for both material are shown in the figure 4.



**Figure 4.** The results of the Vickers microhardness measured on the Watts-Ford specimens.

In the case of ECAP the variation of the strength with the number of passes, using route B<sub>C</sub> and C is shown in figure 5 and the fitted parameters are given in table 2. It can be seen that there is a sharp increase in yield stress after the first ECAP pass for both materials. For A1050 aluminum, after the first ECAP pass the yield stress increased by a factor of about 4.2. The flow stress points a saturation value in both routes, and there is no significant difference between the two examined routes. The difference at a given pass is about  $\pm 3$  MPa. The same tendency can be noticed in the hardness values. For the alloyed aluminum, significant difference can be observed between route B<sub>C</sub> and C. For route C the yield stress increased by a factor of 2.7, while in the case of route B<sub>C</sub> increased by a factor of 3.9 after the eighth pass.

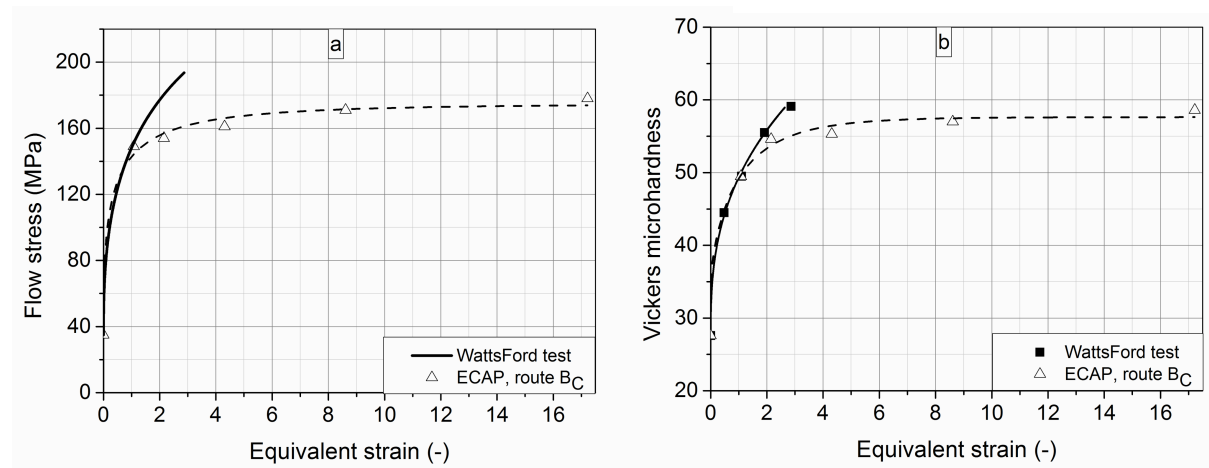


**Figure 5.** The variation of the flow stress for A1050 and Al6082 materials.

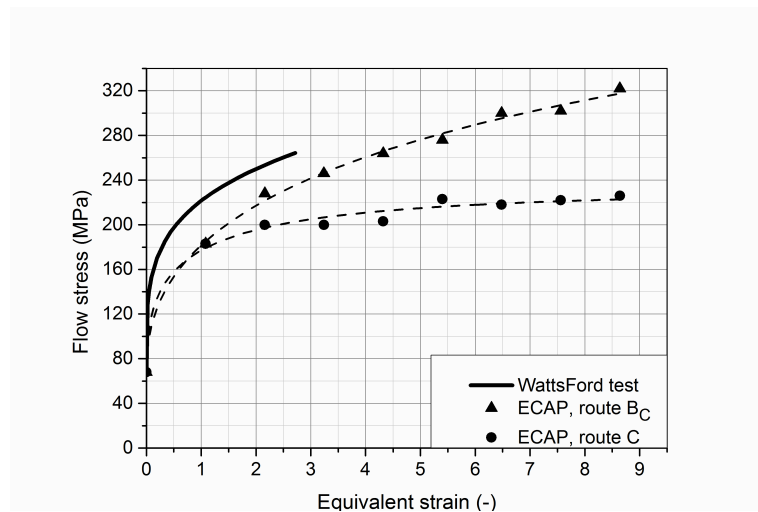
**Table 2.** The parameters of equation (3) for A1050 and for Al6082 processed by ECAP.

| Material                   | $\sigma_0$ (MPa) | $\sigma_1$ (MPa) | $\bar{\epsilon}_c$ (-) | n(-)  |
|----------------------------|------------------|------------------|------------------------|-------|
| <b>A1050</b>               |                  |                  |                        |       |
| route B <sub>C</sub> and C | 35               | 139.7            | 0.41                   | 0.436 |
| <b>Al6082</b>              |                  |                  |                        |       |
| route B <sub>C</sub>       | 68               | 491.03           | 18.2                   |       |
| route C                    | 68               | 162.9            | 0.8                    | 0.46  |

The comparison of the flow stress and hardness of samples produced by Watts-Ford test and by ECAP are shown in figure 6. It can be seen that there is a notable difference in strength and hardness for commercial purity aluminum. The yield stress after sixteen pass (178 MPa) has lower value than that is obtained by Watts-Ford after the plastic strain value of 2.8 (195 MPa). In the comparable plastic strain range such a difference also noticeable. The deviation can be observed in the value of Vickers microhardness. It is noticed that after the first pass, both the hardness and the flow stress values are equal than that obtained by Watts-Ford test. After the subsequent passes the difference increased with equivalent plastic strain. This could be due to the cyclic effect of the ECAP process caused by the rotation of the re-entered billet. The difference in flow stress is noticeable for alloyed aluminum also, although in the case of route B<sub>C</sub> the deviation is smaller. It is shown in figure 7.

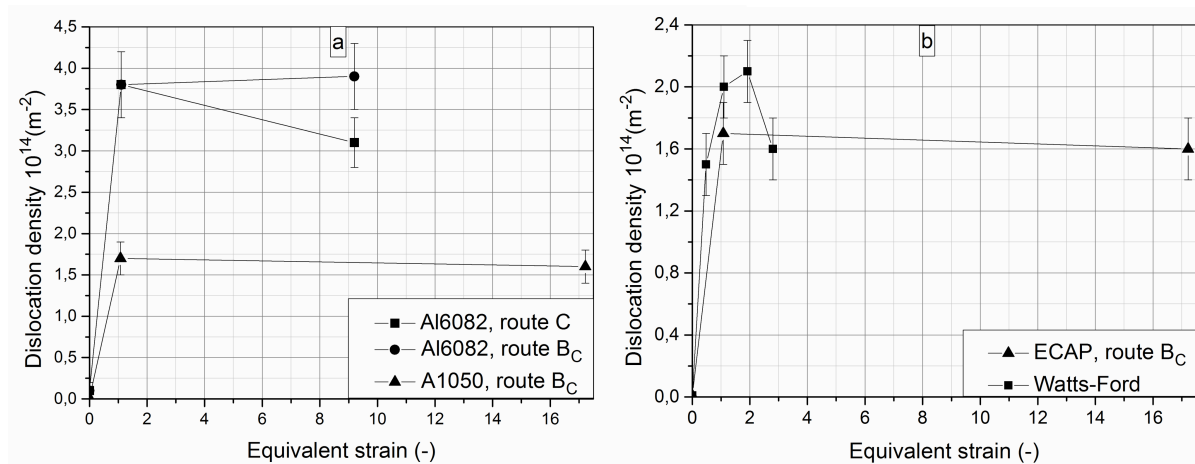
**Figure 6.** Comparison of flow stress (a) and Vickers microhardness (b) obtained by Watts-Ford test and by ECAP for A1050.

It is noticed that the strength and the Vickers microhardness in plane strain compressed specimen is always superior to those in ECAP-processed samples. In Watts-Ford test there is no change in the strain path while in ECAP process the shearing characteristics and so the strain path changes by rotating the sample between each pass. The applied route, with its strong influence on the microstructure and texture, and the change of the strain path together is assumed to cause difference in strength and hardness. For Al6082 processed by ECAP with route B<sub>C</sub> the flow stress is higher after the fourth path. This deviation can be explained by the superior grain refinement effect during ECAP.



**Figure 7.** Comparison of flow stress obtained by Watts-Ford test and by ECAP for Al6082.

For both materials the evolution of the dislocation density can be seen in figure 8(a) after the first and the last pass using route B<sub>C</sub> and C. The ECAP-processed material shows to tend to a saturation value. However, in Al6082 the dislocation density decrease after the eighth pass for route C. These changes can be attributed to the structural relaxation of subgrain boundaries. The decrease of dislocation density can be explained by the annihilation of dislocations. The lower value of dislocation density results lower flow stress values. Figure 8(b) shows that, for A1050, by Watts-Ford test higher dislocation density can be attained in the same strain range than by ECAP. In both process, mainly the edge dislocations are dominant. Materials with high stacking fault energy (SFE) the structure of dislocation is characterized by edge dislocations after room temperature SPD processes. Due to the low SFE, the annihilation of screw dislocations is relatively easy by cross-slip [13]. The area weighted mean crystallite size decreased with strain. However, the crystallite size usually smaller than the grain size obtained by TEM. The coherency of X-rays breaks even if they are scattered from volumes having small misorientation, thus the crystallite size corresponds rather to the subgrain size [14]. The parameters determined by X-ray diffraction profile analysis are summarized in table 3.



**Figure 8.** Evolution of dislocation density for the ECAPed materials (a) and the evaluation of dislocation density in A1050 processed by Watts-Ford method and by ECAP using route B<sub>C</sub> (b).



**Table 3.** The parameters obtained by X-ray line profile analysis <sup>a</sup>.

| Material/Process           | $\langle x \rangle_{\text{area}}$ (nm) | $q^b$ |
|----------------------------|----------------------------------------|-------|
| <b>Al6082</b>              |                                        |       |
| Annealed                   | 500                                    | -     |
| ECAPx1                     | 137±15                                 | 0.4±1 |
| ECAP B <sub>C</sub> x8     | 149±15                                 | 0.4±1 |
| ECAP Cx8                   | 164±15                                 | 0.4±1 |
| <b>Al1050</b>              |                                        |       |
| Annealed                   | 600                                    | -     |
| ECAPx1                     | 166±19                                 | 0.4±1 |
| ECAP B <sub>C</sub> x16    | 154±17                                 | 0.4±1 |
| Watt-Ford 1 <sup>c</sup> . | 171±18                                 | 0.4±1 |
| Watt-Ford 2.               | 160±15                                 | 0.5±1 |
| Watt-Ford 3.               | 145±15                                 | 0.4±1 |
| Watt-Ford 4.               | 119±13                                 | 0.4±1 |

<sup>a</sup> The diffraction profiles was evaluated by the CMWP, Convolutional Multiple Whole Profile fitting procedure.

<sup>b</sup> Characterizing the type of dislocation.

<sup>c</sup> The numbers denote the deformation zone (see figure 2)

The  $q$  parameter is the dislocation contrast factor and depends on the type of dislocation. For face-centered cubic lattice the value of  $q$  is in the range of 0.36-1.33. The lower value denotes edge dislocation, the higher value denotes screw dislocation. The results obtained by X-ray line profile analysis shows that the prevailing type of dislocation is edge dislocation for all of the examined processes.

#### 4. Conclusions

In the present study, the influence of the deformation process on the strength was investigated using commercial purity and alloyed aluminum. The evolution of the dislocation density, flow stress and the Vickers microhardness were examined on the samples processed by Watts-Ford method and by ECAP using route B<sub>C</sub> and C. The following main conclusions can be drawn.

- By monotonic deformation processes higher flow stress values can be achieved than by non-monotonic cyclic severe plastic deformation. This observation is confirmed by the Vickers microhardness measurements. The differences are noticeable in the comparable plastic strain range. The deviation can be explained by the frequent strain path change of ECAP process.
- In the case of alloyed aluminum the deviation in strength is smaller than that obtained by pure aluminum. This can be attributed to the strengthening effect of solid solution.
- The dislocation density points a saturation value in the early number of ECAP passes, and the X-ray line profile analysis showed that the dislocation density is higher in samples produced by Watts-Ford method.
- In samples produced by ECAP and by Watts-Ford test mainly the edge dislocations are dominant due to the annihilation of screw dislocation by cross-slip.

## Acknowledgment

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