Effects of high straining in copper strips processed by multiple direct extrusion and subsequent rolling

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Abstract. As a novel severe plastic deformation (SPD) method, multiple direct extrusion (MDE) has been proposed as a new technique based on conventional deformation, consisting in repetitive extrusion processes of initial-squared cross-sectional workpieces, followed by transversal cutting, overlapping the resulting halves and resuming the extrusion by reinserting samples in the same die. Four cycles of MDE (route B) were applied to commercial copper and the potential for grain refinement and improvement in mechanical properties were evaluated. Because after the 3rd pass MDE (route A) a buckling instability was noticed, a series of comparative experiments by simple and combined rolling (R) and MDE, respectively, were undertaken. Tensile testing conducted at room temperature using a computer-controlled testing machine was carried out to evaluate the mechanical properties after each MDE and/or R cycle. Tensile strength and elongation at fracture after MDE, R and MDE + R, respectively, were analysed. It was found that after MDE cycle (route B), tensile strength is more than two times higher than that of the initial state and the elongation at fracture decreases not less than 25%. Combining MDE and R does not give a new increasing in strength that remains almost the same but decreases elongation at fracture.

1. Introduction
If a deformable conventional metallic material is subjected to a high-straining process its microstructure changes dramatically, due to the grain fragmentation [1]. During this process the coarse grain (25-150 µm) becomes an ultra-fine one (100 nm - 1 µm) with beneficial consequences on the properties [2]. There are some approaches - known as severe plastic deformation (SPD) techniques - to realize grain fragmentation, such as equal channel angular pressing [3,4], cyclic extrusion-compression [5], high pressure torsion [6,7], accumulative roll-bonding [8], repetitive corrugation straightening [9] and multi-axial compression/forging [10]. Among these methods the most investigated remains ECAP which seems to be very efficient for grain refinement. However, as noted by the inventor himself Vladimir Segal, the process of commercialization at large scale has not yet happened [4]. To increase the interest for industrial applications novel ideas-based on the principle of already traditional ECAP process - were reported: ECAP using dies with movable walls to reduce friction [11], ECAP-conform [12], simple and double-billet incremental ECAP [13] and non-ECAP (NECAP) [14].

The extrusion process has a large potential for grain refinement, so the conventional direct extrusion with high ratio in one/two steps [15, 16] and hydrostatic extrusion with extra-high ratio [17] have been investigated, too. More precisely, in high ratio extrusion process, the grain which starts at an initial size of 100 µm, reaches approximately 5 µm after one step and about 2 µm after two steps.
However, some practical issues such as the large deformation force, high pressure in dies, and heating due to the thermal effect of deformation are common phenomena during high ratio extrusion, making this technique difficult to use. Multiple direct extrusion (MDE) was earlier proposed [18,19] as a new technique in grain refinement. A repetitive indirect extrusion technique, known as accumulative back extrusion, has been also described recently [20,21].

The MDE process is a repetitive procedure, but unlike well-known SPD processes, the workpiece changes its cross-sectional shape during deformation. The initial billet (square shaped in cross-section) is extruded several times through a rectangular die aperture. The output of the die has the side as submultiple (half, third, fourth and so on) of the entrance side, so the extrusion ratio per pass is 2, 3, and 4 corresponding to 50%, 66.6%, and 75% reduction, respectively.

The principle of MDE for a 50% cross-section area reduction is depicted in figure 1. MDE starts by extruding the workpiece until the punch arrives near the deformation zone. Then the process stops, the punch is removed and a new billet is inserted into the container to push out the previous billet that partial remained in the deformation zone.

![Figure 1. Schematic principle of MDE and subsequent rolling.](image)

For 50% reduction, the length of the extruded product doubles compared with the initial billet according to constancy volume law. After the complete extrusion, the product is transversely cut at the output ends (to remove the nonconforming zone) and then at the middle of the length. The two halves of the extruded billet are longitudinally joined to get again a square shape in cross-section, and thus a new workpiece is getting. For the next pass, two processing routes can be used:

- Route A: reinserting in the same longitudinally position;
- Route B: reinserting with 90° rotation around longitudinal axis.

Using route A, the extruded billet will result in a rectangular shape after each pass. Only strips can be obtained by this approach. Choosing the route B will give extruded billets with rectangular or square shapes after even or odd pass, respectively.

The procedure can be repeated several times until the imposed strain is achieved. From this point of view, the number of passes depends of the initial cross-section area size.

The effective (von Mises) strain was calculated, being 0.8/pass. For comparison, the maximum effective strain/pass is 1.15 for ECAP with a 90° die angle and 0.8 for ARB.
For a better understanding, the mechanism of grain refinement based on velocity discontinuities is briefly depicted (figure 2). Let us take an arbitrary point on AC and the corresponding point on BC (figure 2a). Because of the continuity condition in material flow, the normal components of velocity across the boundary of the two regions must be equal, so \( (v_{ln} = v_{wn}) \) \( AC \) and \( (v_{lln} = v_{wn}) \) \( BC \). Parallel to the surfaces AC and BC, the tangential components may have different values: \( (v_{wt} \neq v_{lt}) \) \( AC \) and \( (v_{llt} \neq v_{lt}) \) \( BC \). The differences \( (v_{wt} - v_{lt}) \) \( AC \) and \( (v_{llt} - v_{lt}) \) \( BC \) define the velocity discontinuities, giving rise to tangential stress \( \tau \) [18]. When an elongated and thin enough grain (obtained from a previous MDE pass) crosses one velocity discontinuity surface along which tangential stress exceeds strength in pure shear (\( \tau > k \)), the grain strength will be overcame and the fragmentation process starts (figure 2b). Therefore, the velocity discontinuity surfaces play an important role in grain fragmentation. The higher the number of discontinuity surfaces, the more efficient the fragmentation.

![Kinematically admissible flow field: a) components of velocities on slip lines; b) schematic grain fragmentation mechanism during MDE [18].](image)

In the MDE processing, route A, it was noted that under the described conditions of the experiment after 3 passes the buckling stability of the individual specimens is lost. That because of different strain rates in longitudinal section: higher in the middle of the section and lower to the edges. To avoid buckling, rolling in several steps to the final proposed cross-sectional size is applied, thus combining the two processes, as shown in figure 1. The goal of this study is to evaluate the effects of high straining in copper strips processed by MDE– route A - and subsequent rolling (MDE+R). The paper focuses on mechanical properties evolution - in a comparative manner (MDE 4 passes route B and MDE 3 passes route A + rolling) -after each processing stage to emphasize changes in mechanical properties.

2. Experimental

2.1. Processing copper by MDE and subsequent rolling
Commercial copper (purity 99.5%) was used in this study. The specimens were machined as billet with dimensions \( 20 \times 20 \times 50 \) mm\(^3\). A subsequent annealing at 800°C for 30 minutes followed by water quenching was performed to eliminate the strain hardening from previous metal-working processes and achieve a good workability of the material.

After this treatment, the grain size reaches approximately 150μm. Then the specimens were extruded at room temperature using a hydraulic press (figure 4) with 10 mm/s ram speed. Between cycles, the joined billets were reinserted in the same position without rotation around longitudinal axis
Zinc stearate was used as a lubricant to decrease friction at the workpiece-die interface. After the 3rd pass (figure 3d), samples were rolled through several passes to a thickness of 1 mm (figure 3e).

2.2. Tensile testing

Tensile testing was carried out to evaluate the mechanical properties after each MDE cycle and subsequent rolling. The tensile tests were conducted at room temperature using a computer-controlled testing machine (Instron 3382) with a constant strain rate of $6.7 \times 10^{-3}$ s$^{-1}$ applied throughout the entire test according to ISO 6892-1:2016 recommendations. For each pass, specimens have been tensile tested to determine the tensile strength ($R_m$) and elongation at fracture ($A$). Rectangular cross-sectional specimens were obtained by electro-discharge machining as proportional test pieces, figure 5.

3. Results and discussions

Figure 6 shows the results of the tensile testing performed under above conditions. For a better comparison of the data, the initial state of the material (i.e. fully annealed - A) was plotted on the same coordinate system. One compare rolling (R) with MDE (4 passes route B) and MDE+R (3 passes MDE route A + Rolling), respectively.
Experimental data show that the relationship between yield strength (YS) and ultimate tensile strength (UTS) is well described by YS/UTS ratio. As could be seen from table 1, the YS/UTS ratio is approximately the same for the processes involving rolling (R and MDE+R).

Table 1. Yield strength (YS), ultimate tensile strength (UTS), and elongation at fracture (A) after copper processing.

|        | A     | R     | MDE   | MDE+R |
|--------|-------|-------|-------|-------|
| YS [MPa] | 37    | 209   | 93    | 213   |
| UTS [MPa] | 217   | 409   | 476   | 450   |
| A [%]   | 73    | 9     | 27    | 9     |

For fcc metals like copper, YS is given mainly by work hardening so that explains the higher level of YS for R and MDE+R processes. During a unidirectional deformation process such as cold rolling, an increase in the total strain leads to the formation of layered microstructures and the alignment of the transition bands, grain boundaries and sub-boundaries. This has the result that the normals to the flattened units are rotated until they are approximately parallel to the normal direction of rolling. Thus, the microstructures present at large strains are characterized by ribbon-like subgrains and grains which are highly elongated in the direction of metal flow having a high free dislocations density. Because of that, YS and UTS have higher levels but the elongation at fracture is lower. On the contrary, during the first MDE pass, the initial straining introduces a high dislocation density as cellular substructure. Then in the subsequent passes, these cell formations turn into cell blocks subdivided by dislocation walls. So, the initial grains are highly divided, increasing YS as Hall-Petch relationship describes. In other words, the increase of the YS is due to the ultrafine structure free of dislocations rather than the work hardening as for R or MDE+R processes. That explains the increase of YS compared with the annealed copper.
4. Conclusion

Tensile strength and elongation at fracture after MDE, R and MDE + R, respectively, were analyzed. It was found that after MDE (4 passes route B), tensile strength is more than two times higher than that of the initial state and the elongation at fracture decreases, but not less than 25%, which in fact represents an unexpectedly good result. This is the effect of positioning velocity discontinuities in orthogonal planes from one pass to the next one.

Combining MDE (3 passes route A) and R does not give a new increasing in strength that remains almost the same but decreases elongation at fracture. The YS/UTS ratio increases to the same level due to the highly elongated grains in the direction of metal flow also having a high free dislocations density. That explains the higher tensile strength and a lower elongation at fracture. Therefore, MDE route B remains the most suitable for practical applications.

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