Processing of low Carbon steel by dual rolls equal channel extrusion

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Abstract. This paper introduces a new method of forming for achievement of grain structure refinement by processing in DRECE (Dual Rolls Equal Channel Extrusion) equipment. The DRECE device was developed at the VSB - Technical University of Ostrava. It allows grain refinement in strip plate with dimensions of 58 mm (width) x 2 mm (thickness) x 1000 mm (length). The influence of the number of passes on the mechanical properties and related structure refinement was examined experimentally. The effect of a heat treatment (500 °C/1 h/steady air) on the grain refinement of low carbon steel after severe plastic deformation is analysed. Through this novel technique, the grain structure can be converted into a submicron grain structure.

1. Introduction
Several SPD techniques have been successfully applied to produce bulk UFG materials. UFG materials are quite attractive due to their ultrahigh strength, more than twice of the coarse grained equivalents. The term “ultrafine grained structure” is referring to nanostructure with grain sizes less than 100 nm, and sub-microcrystalline structure with grains between 100 and 1000 nm. In recent years, development of manufacturing processes enabling to obtain ultrafine grained structures in steels have become a worldwide effort. Currently, two main approaches exist for refining ferrite grains down to the ultrafine grained range in bulk steels. While the first group comprises advanced thermo-mechanical processes [1], the approach of the second group employs various severe plastic deformation techniques, including ECAP [2], high pressure torsion (HTP) [3], accumulative roll bonding (ARB) [4] and constrained groove pressing (CGP) [5] to refine the structure by introducing large plastic strain into the bulk material. However, these techniques cannot be used for sheet metal forming.

Plastic deformation generates high dislocation density. The dislocations are usually arranged in specific configurations, which depend on the crystalline structure of the material. This concerns the dislocation walls, accumulation at the barriers of slip planes (shear bands), dislocation loops and dislocation cells, and more. The interaction of dislocations leads to the formation of subgrains with low-angle boundaries [6, 7] (see Fig. 1.). These subgrains may further change when exposed to severe plastic deformation, which can lead also to the formation of high-angle boundaries and thus to further refinement of the original grain (see Fig. 2.).

At present, stabilization of the structure of such materials, in association with optimum heat treatment to preserve their high mechanical properties with unchanged or only slightly reduced ductility, is the subject of very intensive research in important research institutions and universities.

Published experimental results indicate that the use of high or severe plastic deformation of metals makes possible to produce materials with extraordinary physical and mechanical properties, which could be used for production of technologically extremely stressed parts or structures. This naturally leads to reduction of their weight. It turns out that this way of research and possible practical realization of the achieved results is very promising. It is also presented as the future of technological development in the given area. A number of phenomena and relationships, conditions and consequences of effects of internal and...
external forces in the microstructure in potentially suitable metals, subjected to multiple
defonnations by extreme pressures, are far from being adequately investigated. Before it is
possible to start their technological applications in material production, it is necessary to
expand objective knowledge not only through for studying all available information, but
primarily also to demonstrate and confirm them by experimental research.

2. Principle of DRECE processing
The main goal of the paper is a review of current achieved results given by processing of low
carbon steel by the DRECE technology, the prototype equipment of which has been put into
service at the Department of Mechanical Technology at VSB – Technical University of
Ostrava [8]. The paper describes results of determination of the influence of number of
passes on resulting mechanical and microstructural properties of the processed low carbon
steel. The paper proposes testing procedures for the determination of microstructural
stability of ultrafine grained structure (UFG) materials obtained in DRECE process. The
proposed DRECE process belongs to the group of progressive type of forming processes
making use of severe plastic deformation (SPD). The SPD methods make possible to process
metallic materials to achieve UFG structure with mean grain sizes around or less than 1µm.
After this processing step the material exhibits - in comparison with the structure of
conventional materials - significantly better mechanical properties, especially in yield stress
and in fracture tensile strength. The prototype of the DRECE equipment is shown in Fig. 3.

3. Experimental material and procedures
Carbon steel in as cold rolled state, without previous heat treatment, of chemical purity
according to the relevant Czech standard ČSN 41 1321, was used for investigation in the
form of sheet with dimensions of 58 x 2 x 1000 mm³ (see Fig. 4). The chemical composition
of the investigated steel is given in Table 1. The carbon steel sheet strips were extruded
through the DRECE equipment at ambient temperature, without previous heat treatment or operative heating after individual passes:

a) up to 2, 4, 6 passes

b) up to 2, 4, 6 passes followed by heat treatment at 500 °C/1 h/steady air

c) up to 2, 4, 6 passes followed by heat treatment 500 °C/1 h/steady air and then by the 2nd series of DRECE forming for another 2 passes.

The sheets were rotated by 180° around their longitudinal axes between individual passes. Hardness HV10 and mechanical properties: yield stress Rp 0,2, tensile strength Rm and ductility A80, were measured in tensile test in the initial state and also after DRECE processing. The investigation was completed by metallographic evaluation of the microstructure of selected samples.

### Table 1

|          | C | Mn | P  | S  |
|----------|---|----|----|----|
|          | 0.10 | 0.43 | 0.03 | 0.03 |

#### 3.1 Evaluation of dimensional changes of strip after passing through the DRECE equipment

The dimensional changes of the sheet strip after extrusion through the DRECE equipment is shown in Fig. 4. Macro-photos of strip of sheet in initial state and after 6 passes are shown in Fig. 5. The values of dimensional changes after the extrusion process of the sheet after individual passes through the DRECE equipment is shown in Table 2. All dimensions (length-l, width-b and thickness-t) were measured at the places marked in Fig. 4. It is evident from the table that the steel sheet behaves similarly as previously investigated copper and aluminium [8]. The measured length was shortened after six passes by a maximal value of 23 mm, on the other hand, the width increased by 1.4 mm.

| Number of passes | Route B_A [°] | l × w × t [mm] | t1 | t2 | t3 |
|------------------|--------------|----------------|----|----|----|
| Initial state    |              | 300 48.09 48.13 48.09 | 1.90 | 1.91 | 1.89 |
| 1                | 0            | 298 48.64 48.51 48.30 | 2.01 | 1.92 | 1.90 |
| 2                | 180          | 294 48.64 48.54 48.55 | 1.90 | 1.96 | 1.91 |
| 3                | 0            | 290 48.82 48.89 48.75 | 1.90 | 1.98 | 1.93 |
| 4                | 180          | 285 48.92 49.08 48.90 | 1.92 | 1.99 | 1.98 |
| 5                | 0            | 280 48.99 49.28 49.14 | 1.86 | 2.01 | 1.98 |
| 6                | 180          | 277 49.08 49.58 49.37 | 1.83 | 2.04 | 2.03 |

Fig. 4: Dimensions of the strip sheet with marked places used for measurement of width, thickness and length

Fig. 5: Photo of the sheet strips from low carbon steel (a-initial state, b-after 6 passes through the DRECE equipment).
3.2 Vickers hardness
Hardness evaluations (HV10) were performed with use of hardness tester HPO 250 on the cross-section of cut samples at the place of measurement for all series and on all the strips (see Tables 3, 5 and 7).
It is evident from Table 3 that in the case corresponding to the series “a” the average hardness value increased significantly after the 2\textsuperscript{nd} pass and slightly after the 4\textsuperscript{th} pass. This confirms that the number of passes exceeding 4 has no significant influence on the increase of hardness. The achieved highest value 135 (HV10) is higher (approx. by 45 \%) in comparison to the hardness value in the initial state. This confirms the correct functionality of the DRECE equipment.

| Number of passes | Hardness HV 10 (average value) |
|------------------|-------------------------------|
| Initial state    | 93                            |
| 2                | 122                           |
| 4                | 135                           |
| 6                | 136                           |

Table 3 Obtained hardness values corresponding to the series “a”

Table 4 Obtained hardness values corresponding to the series “b”

| Number of passes | Hardness HV 10 (average value) |
|------------------|-------------------------------|
| 2                | 131                           |
| 4                | 131                           |
| 6                | 138                           |

Table 5 Obtained hardness values corresponding to the series “c”

3.3 Evaluation of mechanical properties by tensile testing
The mechanical properties of the low carbon steel (yield stress $R_{p0.2}$, tensile strength $R_m$ and ductility $A_{80}$) were evaluated by tensile tests on the samples taken from selected passes. All tensile tests were performed according to the ISO 6892-1 standards. Tables 6, 7 and 8 summarises the results. The results are also shown in graphical form (see Figs. 6 - 8).
For the case corresponding to the series “a” the results are given in Table 6 and Fig. 6. It can be seen from the table and from the diagram that values of the yield stress $R_{p0.2}$ and of the tensile strength $R_m$ increase significantly already after the second pass. This is in good agreement with the hardness tests. The increase is then gradual with a maximal value of $R_{p0.2} = 390$ MPa. The maximal value of tensile strength is $R_m = 415$ MPa.
This increase represents already after the 4\textsuperscript{th} pass an increase of $R_{p0.2}$ by approx. 125\% and of $R_m$ by approx. 33\%. The results after the 6\textsuperscript{th} pass exhibit almost comparable values – this is in good agreement with the performed hardness tests as mentioned above. However, the ductility of the material decreases rather significantly. It is dropped from the initial value of 50.3\% down to the lowest value of 14.8\% after the 6\textsuperscript{th} pass. The slight increase of ductility after the 6\textsuperscript{th} pass might have been caused by recovery processes.
**Table 6** Mechanical properties for the series “a”

| Number of passes | \( R_{p0.2} \) [MPa] | \( R_m \) [MPa] | \( A_{80mm} \) [%] |
|------------------|----------------|----------------|----------------|
| Initial state    | 173            | 311            | 38.3           |
| 2x               | 370            | 391            | 22.6           |
| 4x               | 383            | 411            | 15.8           |
| 6x               | 390            | 415            | 14.8           |

**Table 7** Mechanical properties for the series “b”

| Number of passes | \( R_{p0.2} \) [MPa] | \( R_m \) [MPa] | \( A_{80mm} \) [%] |
|------------------|----------------|----------------|----------------|
| 2x               | 243            | 334            | 17.8           |
| 4x               | 250            | 335            | 18.8           |
| 6x               | 254            | 335            | 17.8           |

**Table 8** Mechanical properties for the series “c”

| Number of passes | \( R_{p0.2} \) [MPa] | \( R_m \) [MPa] | \( A_{80mm} \) [%] |
|------------------|----------------|----------------|----------------|
| Initial state    | 173            | 311            | 50.0           |
| 2x               | 401            | 456            | 7.2            |
| 4x               | 409            | 454            | 5.4            |
| 6x               | 395            | 447            | 5.6            |

**Fig. 6:** Influence of number of passes through the DRECE tool on yield stress \( R_{p0.2} \)
Fig. 7: Influence of number of passes through the DRECE tool on tensile strength $R_m$

Fig. 8: Influence of number of passes through the DRECE tool on ductility $A_{80}$
3.4 Metallographic analysis

The evaluation of mechanical properties was completed by metallographic analysis of the microstructure of the investigated low-carbon steel after individual passes through the DRECE tool examined by light microscope NEOPHOT 2.

After usual metallographic preparation the test-pieces were etched in NITAL. The observed microstructures of low-carbon steel in the initial state and on the samples taken from cross-section after selected passes are shown in Figures 9-13. The microstructure of the investigated steel in its initial state is formed by slightly elongated ferrite grains and by fine pearlite particles [8].

As it is evident from the photos of the microstructures for the series “a”, no significant refining of the structure occurred during the DRECE process. The average grain size was determined in accordance with the EN ISO 643 as G 8-9 standards. All evaluated samples of the steel showed this value after the 2nd, 4th and 6th pass on cross-section. The grain structure was influenced the most significantly after the 2nd pass, where the average size from the initial state was \( \bar{d} = 60 \, \mu m \) and after the 2nd pass approx. \( \bar{d} = 15 \, \mu m \). After the 6th pass a grain size of 10 \( \mu m \) was achieved. This steel does not contain sufficient volume of admixtures from creation of precipitates, which would enable greater structure refinement.

9: Structure - initial state (mag. 500×)  

10: Structure after the 2nd pass (mag. 500×)
3.5 Influence of heat treatment on mechanical properties
The main goal of the heat treatment was to homogenize the structure which led to partial loss of the strength values.
The heat treatment conditions were selected based on the material properties and on the expected final properties. The strips processed by the above mentioned DRECE forming process were heat treated in order to see the effect of applied heat treatment on basic mechanical properties.
The results given in this paper are only partial. The influence of the second series of DRECE process after previous one and after applied heat treatment was also evaluated. The evaluation of basic mechanical properties was performed in the same way as before.

Fig. 11: Microstructure after the 4th pass (mag. 500×)

Fig. 12: Microstructure after the 2nd pass + HT, series “b”

Fig. 13: Microstructure after the 2nd pass + HT, series “c”
Results of procedures corresponding to the series “b”:
The results concluded from the hardness tests are shown in Table 5. The obtained results exhibit lower hardness values and have approximately the same hardness values between individual passes. The tensile tests were performed under the same testing conditions as applied in previous tests. The results are shown in Table 6 and in Figs. 6-8. The results from tensile test confirmed the observed hardness values. No significant differences between passes exist. The material exhibits increased strength values, as well as relatively good ductility. Thus, the DRECE processing followed by appropriate heat treatment could be a new way to produce low carbon steel strips with advanced mechanical properties.

Results of procedures corresponding to the series “c”:
Part of the samples after the first DRECE processing and following heat treatment was further processed by one more series in DRECE forming in order to evaluate its influence. The results from hardness tests are shown in Table 5. The obtained results exhibit lower values of hardness and have approximately the same hardness values between individual passes. The tensile test was performed under the same testing conditions as applied in previous tests. The results are shown in Table and in Figs. 6-8. As it can be seen from Table 7, the measured hardness values are somewhat higher than those corresponding to the series “a” and the increase of this value depends on the total number of passes. $R_{p0.2}$ shows a similar increase as that corresponding to the series “a” and its value is slightly higher. The ultimate strength $R_m$ shows similar increase, but its values are significantly higher than those corresponding to the series “a”.

Results of metallographic analysis corresponding to the series “b” and “c”:
As it is evident from the photos of the microstructures shown in Figs. 12 and 13 of the investigated low-carbon steel, no significant refining of structure occurred during the DRECE process.

3.6 TEM analysis
The metallographic evaluation of the microstructure of the samples in initial state without heat treatment and after application of the ECAP process was complemented by studying the substructure using TEM (Philips CM20 equipment). The results of the substructure of the steel sample in the initial state and after selected passes through the DRECE device are shown in Figure Fig. 14 (initial state), Fig. 15 (after the 1st pass), Fig. 16 (after the 3rd pass), Fig. 17 (after the 5th pass) and Fig. 18 (after the 6th pass).
It is possible to assess from the above figures that the substructure formed by an irregular distribution of dislocations in the initial state and after the first pass changes and after the following passes Fig. 16 (after the 3rd pass), Fig. 17 (after the 5th pass) and Fig. 18 (after the 6th pass) shows probably occurrence of micro-bands with their more regular distribution. Their number increases with the increasing number of passes.

On the basis of these observations of substructure it is further possible to assess the results of changes in the properties of the steel after heat treatment and after subsequent application of the DRECE process. Study of changes of the substructure in such cases will be addressed in future works.

4. Conclusions
This paper introduces a completely new method of forming, allowing the grain refinement by use of the process DRECE (Dual Rolls Equal Channel Extrusion).
The device was developed at the VSB - Technical University of Ostrava. A prototype equipment was designed for production of UFG structure in form of strip low carbon sheet steel with thickness of 2 mm.
The obtained results show that the equipment is fully functional.
The experimental investigation of the microstructure refined by the DRECE process have confirmed the suitability of this technology for production of UFG structure in low-carbon steel, which leads to substantial increase of its mechanical properties. Substantial increase of yield stress $R_{p0.2}$ and ultimate strength $R_m$ was achieved, which opens up much broader
possibilities of its application for manufacturing of high strength machine components (since these values in the given steel grade increased). This increase in mechanical properties cannot be achieved by conventional methods of rolling of alloys as discussed in [9].

The strips processed by the above mentioned DRECE forming were heat treated in order to observe the influence of the used heat treatment on the initial mechanical properties.

The main goal of the experiment was to find whether the heat treatment can be used like an interstate process between two series of DRECE forming. The evaluation of the mechanical properties was completed by metallographic evaluation of the microstructures of the investigated low-carbon steel sample after individual passes through the DRECE tool on light microscope NEOPHOT 2. As it is evident from the above photos of microstructures of the investigated low-carbon steel no significant refining of structure occurred during the DRECE process.

It is possible to assess from the above figures that the substructure formed by irregular distribution of dislocations in the initial state and after the first pass changes. After the following passes probably occurrence of micro-bands with irregular dislocation distribution in individual strips is occurred. Their number increased with the increasing number of passes.

The results given in this paper give only partial information about the influence of applied heat treatment and further investigation will be required to modify the heat treatment. From these results it is evident that suitable heat treatment could influence further increase of yield strength.

The equipment DRECE is still at the stage of verification and future works will be focused on verification of the influence of technological parameters on the increase of efficiency of SPD process for obtaining of UFG structure in new types of ferrous metals.

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