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The influence of post-deformation annealing temperature on the mechanical properties of low-carbon ferritic steel deformed by the DRECE method

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The paper presents observations on the mechanical properties characterizing low-carbon steel subjected to deformation by the dual rolls equal channel extrusion (DRECE) method and annealed for 60 min in different temperature variants in the range of 450–700°C. The DRECE process was carried out up to seven passes at ambient temperature. The investigations carried out revealed that the strength of the steel strips increases corresponding to the rise in the number of DRECE passes applied. The yield strength (YS) after seven passes is >2.5 times higher compared to the material in the initial state (before the deformation process). However, the tensile ductility decreased significantly after the DRECE. In order to obtain favorable mechanical properties, the steel strips were subjected to annealing. Our study demonstrates that after being processed by the DRECE method, low-carbon steel can be subjected to low-temperature annealing to ensure that it is endowed with high strength, while maintaining the characteristic good ductility of the material. The results of the research were analyzed in the context of an investigation into the microstructure change, assessed by scanning transmission electron microscopy (STEM), induced in low-carbon steel subjected to the DRECE process and low-temperature annealing.

Keywords: *severe plastic deformation, mechanical properties, low-carbon steel*

1. Introduction

An interesting phenomenon is the dependence of the mechanical properties on the annealing temperature in ultrafine-grained (UFG) [1] and nanostructured materials [2] obtained by severe plastic deformation [3]. This issue may be of special interest for researchers dealing with questions related to high-strength and ductility in UFG materials [4] and nanomaterials [5]. Therefore, it is important to confirm the influence of the annealing process on the mechanical properties of low-carbon steel [6] deformed using severe plastic deformation methods, and this confirmation can be obtained through static tensile tests [7]. One of the methods of SPD is hybrid dual rolls equal channel extrusion (DRECE) method [8]. This method involves repeated passes of steel strips between a set of dies, with angle formation defined by the shaping tool. The multiple deformation affects the refinement of the microstructure and thus causes a related improvement in mechanical properties compared to the initial state of the material [9]. A detailed description of the DRECE method can be found in Rusz et al. [10]. The DRECE method is successfully used for forming low-carbon steel strips [11] due to the possibility of forming material with much larger sections than is possible using the classic SPD methods, and since deformation technology can be deployed without reduction in material thickness [9]. Some properties of low-carbon steel after the DRECE process have already been investigated. The increase in strength properties of steel after successive passes has been confirmed [9]. However, the limited tensile ductility of low-carbon steel strips processed by the DRECE method requires improvement with low-temperature annealing after plastic deformation, and the improvement needs to be carried out without any consequential significant decrease in strength [10]. Wang and Ma [12] confirm that the ductility of UFG materials [13], in particular non-ferrous metals and their alloys [14], may be effectively improved through

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the application of low-temperature annealing after using the classic methods of severe plastic deformation [15]. However, there is a need to ascertain the influence of post-annealing on the mechanical properties of low-carbon steel deformed by the unconventional and hybrid DRECE method. So far, this subject has been mentioned only in a few scientific papers. The results of the experimental studies comprised in this research will allow us to determine whether, in the case of low-carbon steel, heat treatment can be used as an inter-operative process between the individual DRECE passes.

This study presents the results of static tensile tests of low-carbon steel deformed by an unconventional SPD method, before and after annealing processes carried out at various temperatures. The static tensile test results are analyzed in the light of the results obtained from an investigation of microstructure change in the low-carbon steel after annealing; the assessment of the microstructure was carried out using scanning transmission electron microscopy (STEM).

2. Material and experimental procedures

Investigations were performed on ferritic low-carbon steel with a chemical composition (wt.%) of 0.12% C, 0.23% Si, 0.26% Mn, 0.02% Cr, 0.03% Ni, and Fe the balance.

The samples were in the form of steel strips with a length of 600 mm, width of 60 mm, and thickness of 1.9 mm. The material was extruded by the DRECE method at room temperature up to seven passes with the channel angle $\alpha = 108°$ [9]. The steel strips' orientation was kept the same for each pass. The time and temperature ranges of post-deformation annealing were selected based on the results of the experimental research forming part of Kowalczyk et al. [16].

Static tensile tests were carried out on a ZWICK testing machine as required by the specifications of the ASTM standard [17]. After the completion of the DRECE process and the post-deformation treatment, the tensile specimens were cut from the steel strips, parallel to the rolling direction. Three tensile samples for each DRECE pass variant were extracted, having a gauge-length of 30 mm and width of 8 mm.

The details of structural evolution in the samples were observed using STEM (Hitachi HD-2300A). The samples for STEM observations were prepared by mechanical grinding, followed by electrolytic polishing using a TenuPol-5 device by Struers working at a voltage of 43 kV for 20 s. The observations of the fracture surfaces of the samples were carried out using a Hitachi S-4200 scanning electron microscope (SEM).

3. Results and discussion

3.1. Mechanical properties

In order to obtain high strength while maintaining the good ductility of low-carbon steel, the DRECE-processed material needs to be subjected to low-temperature annealing; thus, after the seventh pass of DRECE processing, the steel strips were annealed in the temperature range of 450–700°C for 60 min. Table 1 shows the mechanical properties of SPD-processed steel strips, and the same properties after annealing. Figure 1A shows the change in stress–strain curves after seven DRECE passes, and for the annealing temperatures of 550°C and 650°C. The change in total elongation is indicated in Figure 1B.

From Table 1, it can be seen that annealing after DRECE deformation at 450–550°C has no significant influence on the strength and ductility combination, which indicates that the phenomenon of recrystallization is not observed in the material. With the gradual increase in elongation to the annealing temperature of 600°C, a decrease in the tensile strength value is also noticed. This means that the deformed microstructure is dominated by the phenomenon of recovery with partial-recrystallization.

It can be seen from Figure 1 that the annealing at temperature >600°C leads to a decrease in tensile strength, while increasing the total elongation of the investigated steel. The tensile strength, yield strength (YS), total elongation, and uniform elongation (UE) after annealing at 650°C were determined, respectively, as 436 MPa, 415 MPa,

Table 1. Mechanical properties of steel strips after selected passes in the SPD and annealing processes; the results are presented as measurement ± expected error

Condition	UTS, MPa	YS, MPa	TEL, %	UE, %
As received	314 ± 6	180 ± 4	47 ± 4	20 ± 2.0
After 1st DRECE pass	393 ± 10	385 ± 8	14 ± 4	1.2 ± 0.1
After 3rd DRECE pass	415 ± 6	390 ± 8	12 ± 3	1.1 ± 0.1
After 7th DRECE pass	495 ± 3	487 ± 9	9 ± 5	1.1 ± 0.1
After annealing at 450°C	479 ± 5	462 ± 6	14 ± 2	2.0 ± 0.7
After annealing at 500°C	470 ± 3	459 ± 5	14 ± 4	2.4 ± 0.6
After annealing at 550°C	459 ± 4	448 ± 3	16 ± 4	2.8 ± 0.9
After annealing at 600°C	451 ± 4	428 ± 7	21 ± 3	5.1 ± 1.0
After annealing at 650°C	436 ± 2	415 ± 3	29 ± 2	7.5 ± 1.5
After annealing at 700°C	340 ± 6	188 ± 4	41 ± 2	15.7 ± 1.2

DRECE, dual rolls equal channel extrusion; TEL, total elongation to failure; UTS, ultimate tensile strength; UE, uniform elongation; YS, yield strength.

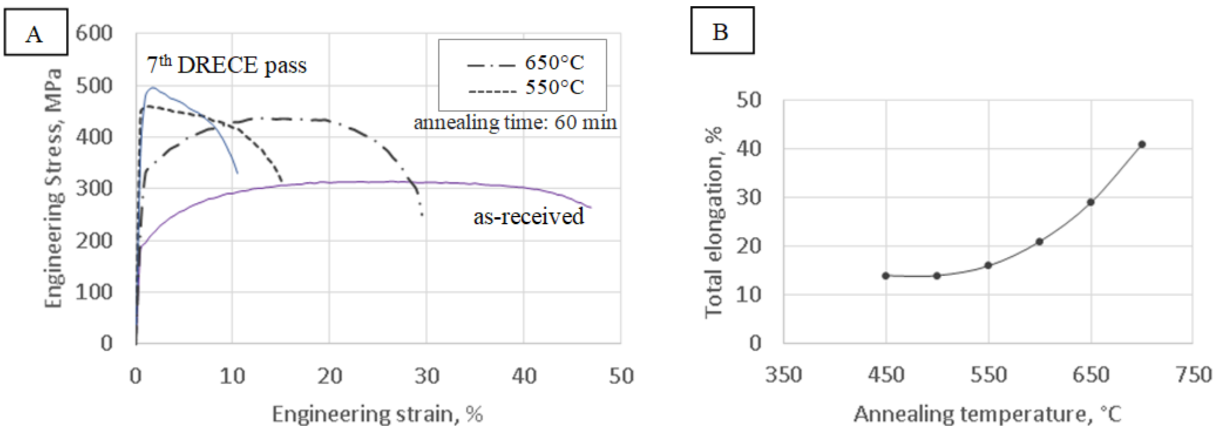

Fig. 1. **(A)** Comparison of the stress–strain curves of the DRECE-processed low-carbon steel strips; **(B)** The curve depicting the change in total elongation against annealing temperature. DRECE, dual rolls equal channel extrusion

29%, and 7.5%. These results imply that low-temperature annealing of low-carbon steel strips after severe plastic deformation by the DRECE method allows us to obtain high strength while maintaining the good ductility of the deformed material.

3.2. Microstructural evolution

The static tensile tests showed that the low-carbon steel processed using SPD and subjected to annealing was characterized by increased strength and ductility [18]. The changes in the mechanical properties of low-carbon steel after SPD and annealing, as indicated by the static tensile tests, correlate with the changes in the microstructure of the material as observed by the STEM technique, as also with the results of fracture surface analysis carried out via SEM. Changes in the microstructure observed after seven DRECE passes and annealing in different temperature variants are shown in Figure 2A–D.

The microstructure of the as-deformed sample exhibits fairly equiaxed ultrafine grains with inadequately developed grain boundaries (Figure 2A).

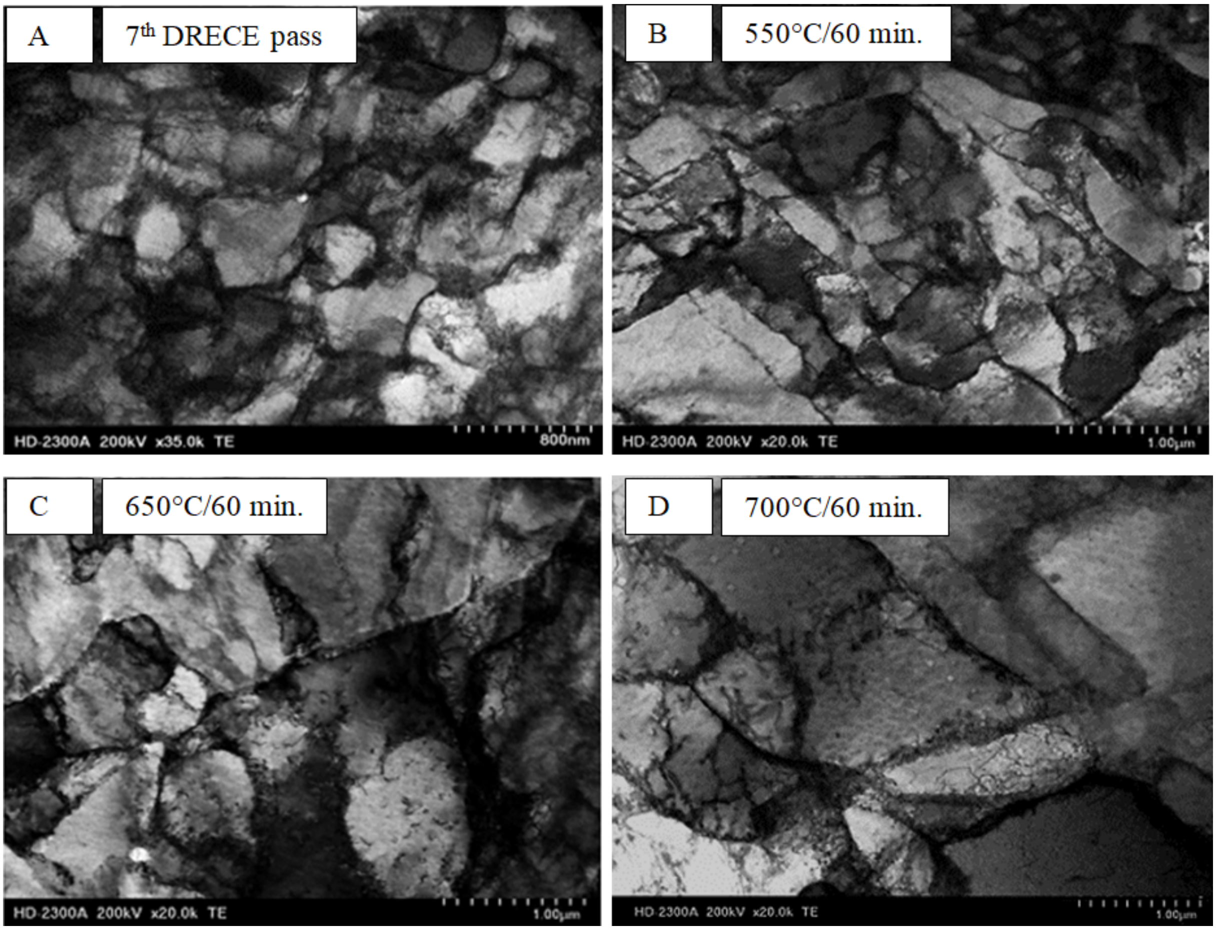

Fig. 2. STEM photographs of low-carbon steel annealed for 60 min in different temperature variants after seven DRECE passes; deformed sample **(A)**, annealed at 550°C **(B)**, 650°C **(C)**, 650°C **(D)**. DRECE, dual rolls equal channel extrusion; STEM, scanning transmission electron microscopy

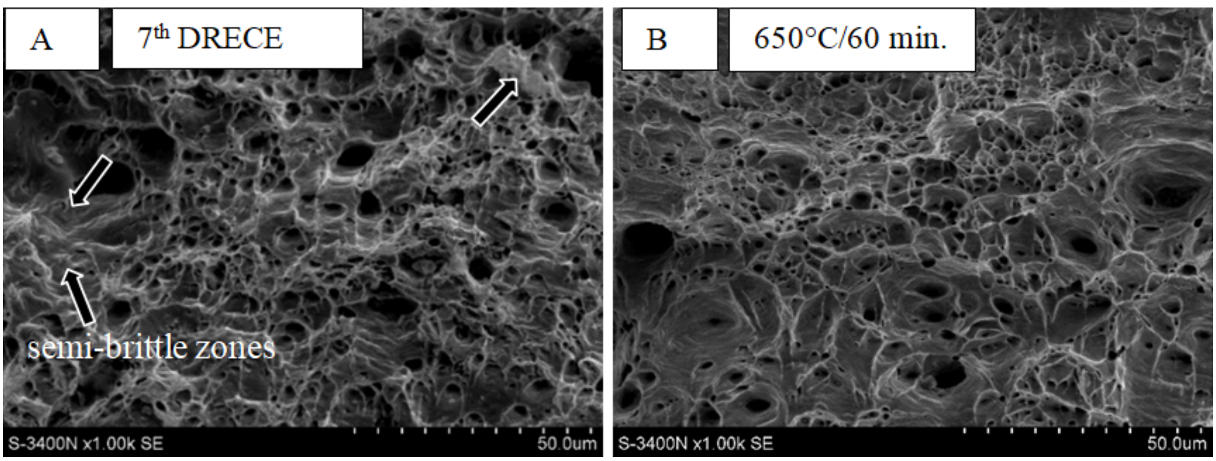

Fig. 3. Fracture surfaces of samples after static tensile test: **(A)** after seven DRECE passes **(B)** annealed at 650°C. DRECE, dual rolls equal channel extrusion

This microstructural characteristic of the ferrite-phase may indicate that the grain boundaries are in the nonequilibrium conditions, with high internal stresses. The microstructural research of the sample annealed at 550°C (Figure 2B) shows some interesting features in comparison with the microstructure of the post-deformation sample after seven DRECE passes: a slight grain growth was noticed, the dislocation density inside an individual grain had become low, and almost all grain boundaries were well defined. The observation of the microstructure of the sample annealed at 650°C may indicate that the formation of a well-defined boundary can be related to the recovery process as a result of absorption of dislocations by the grain boundaries. These observations suggest that recrystallization became active (Figure 2C). The annealing >650°C causes recrystallization. As shown in Figure 2D, in the sample annealed at 700°C, significant growth of the grain has occurred.

Fractographic examinations of the samples are presented in Figure 3A and 3B. On the fracture surface of the sample obtained after seven DRECE passes, characteristic pits typical of ductile fractures are observed; however, small semi-brittle-zones are also visible (Figure 3A). Parabolic holes and numerous pits can be observed on the fracture surface of the sample obtained after annealing at 650°C; this indicates that the fracture surface is ductile (Figure 3B).

4. Conclusions

A UFG low-carbon steel obtained by DRECE was annealed at temperatures in the range of 450–700°C and the influences of annealing on the microstructure and mechanical properties were examined. The following are the main conclusions that can be drawn from this research:

1. Low-carbon steel in the form of strips was successfully processed using the DRECE method up to seven passes at room temperature. However, the limited ductility of the processed steel strips is a potential concern, and this can be addressed by applying a relevant low-temperature annealing after deformation, without significant decrease in tensile strength.
2. Annealing at 600°C caused a progressive decrease in tensile strength and increase in the total ductility. Recrystallization and grain growth occurred at 700°C, which in turn led to a significant decrease in strength and recovery of ductility in the investigated material. The tensile strength, YS, total elongation, and UE after annealing at 650°C were defined, respectively, as: 436 MPa, 415 MPa, 29%, and 7.5%. These parameters indicate that annealing at 650°C results in achieving a good combination of strength and ductility in the low-carbon steel strips processed by DRECE.
3. At the annealing temperature of 600–650°C, the change in the microstructure of the investigated steel was dominated by recovery, which was related to the absorption of dislocations by the grain boundaries. The microstructure after annealing at 700°C is similar to the conventional recrystallized structure.
4. Based on research into the fracture surface topography of low-carbon steel subjected to the DRECE process and annealed at 650°C, it was concluded that the identified areas of holes with various sizes and the parabolic shape of the pits formed as a result of combining microcavities indicate the typical ductile fractures.

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