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Continuous severe plastic deformation of low-carbon steel: physical-mechanical properties and multi-scale structure analysis

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Abstract

A successful attempt to incorporate the advantages of severe plastic deformation (SPD) methods in the continuous drawing process for low-carbon steel is demonstrated. The structural features are considered on different scale levels, using a wide range of methods. While combining shear deformation, which parallels the basis of SPD with the conventional scheme, the cyclic process of grain refinement could be reached. As a result, the plasticity becomes enhanced. At the same time, an important characteristic such as residual stress also has a positive influence on manufacturability; particularly, the existence of the compression stress after shear deformation. The peculiarity of the structure affects the behavior of both mechanical and physical properties (like density, plasticity). The application of drawing with shear (DSh) technology as based on SPD principles, the mechanical softening effect is observed, as is the healing of micro-voids. Such positive affection gives the opportunity to increase the effectiveness of drawing technology through controlling plasticity (ductility). Additionally, it is considered exhaustion of the plasticity resource (EPR). It is shown that in the case of multi pass deformation, there is a parabolic dependence of the EPR measurement, and minimum damage is achieved using a specific combination of partial reductions.

Keywords: continuous severe plastic deformation; drawing; low-carbon steel; structure; properties;

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1. Introduction

Drawing is one of the most widespread methods of metal forming processes [1–3], and welding wire is used in lots of construction and production processes. Thus, the technology of wire manufacturing is very important. Notably, partial reduction [4] during wire drawing, in the mean of exhausting of plasticity resources, is essential. Good optimal reduction choice is a very important step in developing the effective manufacturing technology of drawing. When the plasticity resource is exhausted during the drawing process [4], the wire starts breaking and the whole process is stopped. Then–additional softening annealing is needed. The main aim of this procedure is to heal microvoids and eliminate strain hardening. However, this is a very expensive procedure. In addition, there is a need to enhance the performance characteristics of drawn steel for a minimum number of intermediate softening annealing steps [5]. Severe plastic deformation (SPD) methods are able to solve these tasks.

Today, SPD enables the obtaining ultrafine-grained (UFG) structure in almost any material [6–9]. These materials possess unique and complex mechanical properties, such as a combination of high strength and plasticity [7,10,11]. The main features of the SPD [12] is an accumulation of a high degree of strain. This is possible, due to the shape of the billet that remains unchanged from pass to pass (i.e., Brigman anvils, equal channel angular pressing, twist extrusion). During these metal forming processes, the dominant deformation scheme is simple shear. This peculiarity of SPD methods was at the basis of the continuous process design [13]. While manufacturing wire with SPD methods, one of the limitations is that it occurs in a continuous drawing scheme. A typical volume of manufacturing is calculated in terms of tens of thousands of kilometers and in tons. Therefore, when incorporating SPD into the drawing process, special dies should be used without decreasing the process efficiency.

There is much research aimed at SPD methods of application in drawing [14–18]. The simplest in terms of implementation is the method described in [14]. Authors consider the use of reverse bending on cold drawn fittings without additional heating. The main advantages of this method are its continuity and its ability to be used to produce long products with enhanced mechanical properties. Another SPD method for producing long products combined with drawing is described in [15]. Its main advantage is the continuity

of the process and the possibility to apply it for drawing mass production. The disadvantage of this method is the process complexity during drawing as it uses a complex technical assembly, which needs to be dismantled and reassembled when changing the dies. In Ref. [16], it is shown that the most effective SPD technique is to bend the wire during the drawing. This can be achieved, for example, by changing the location of dies relative to the drawing axis. The main disadvantages of such to this scheme are low speed (0.05 m/s), the difficulty of filling wire into the die before each new drawing, and the complex system of the die unit, which can significantly reduce the drawing efficiency if it breaks. These methods show that the change of metal flow is relative to the axis during drawing and leads to positive effects on the structure and properties. However, some of them showed conflicting information about the change in the mechanical properties of obtained wire [14].

It is of great importance to consider the factors that affect the efficiency of drawing while developing a technology based on the SPD process (continuous SPD). These include the speed of wire passage through the die, the required number of passes, and the manner of dies combination. Optimization of these parameters will allow creating a steel wire drawing technology with a particular required efficiency and manufacturability. Significant improvements in efficiency could be reached with the elimination of intermediate annealing. For this, the enhanced plasticity resource (PR) is needed. Measuring damage to the physical density includes a lot of constituents. These features should be studied from microstructure to properties at each stage. Thus, the purposes of this article are 1) to study the effect of drawing with shear both on the microstructure and on the mechanical properties, of low-carbon steel with various combinations of standard round dies and dies with shear and 2) to create a new technology of drawing with the shear of low-carbon wire, which will eliminate intermediate annealing.

2. Methodology

The studied material is a commercial low-carbon steel (0.08 C, 1.87 Mn, 0.82 Si, 0.02 S, 0.022 P wt% - grade G3Si1, most widespread welding wire).

2.1. Deformation scheme

The drawing with shear (DSh) was carried out by using a specially designed set of dies which promoted the "twist" character of the material flow. A schematic sketch of the twist die channel is shown in Figure 1. To estimate the equivalent true strain during drawing with shear, a simplified relation in the form of the equation below was used as in [19]:

$$e = 2 \cdot \ln\left(\frac{d_0}{d}\right) + \frac{\tan(\beta)}{\sqrt{3}} \quad (2.1.1)$$

Fig.1. Schematic illustration of the shear dies channel (a) - sketch, (b, c) – 3D model. Equivalent strain distribution (Finite Element Method (FEM) modeling) for a cross-section for drawing (d) and DSh (e)

where d_0 and d are the wire diameters before and after drawing, respectively, and $\tan(\beta)$ is the tangent of the angle of the twist line slope (25° or 0.44 rad), as defined in Figure 1 a. In this equation, the first term is the normal drawing strain and the second represents the “twist” component of strain [20]. Based on the above equation, the material underwent ~ 0.25 of “twist” strain in each drawing pass. However, it should be emphasized that these

two strain components have completely different natures and thus Eq. (2.1.1) is oversimplified.

To show the effect of such die geometry finite element method simulations of deformation during DSh were conducted with the aid of Deform-3D software permitting three-dimensional analysis. Design model of DSh die is shown in Figure 1. The die was modeled with rigid elements, while 50,000 tetrahedral elements were employed for the samples and the adaptive meshing was used to accommodate large strains during simulations [21,22]. Friction between the samples and the matrix walls was expressed according to the Zibel's law [23]: $\tau = \mu \cdot \sigma_y$, where σ_y - yield stress, μ – friction coefficient ($\mu = 0.1$). Equivalent strain distribution for a cross-section for drawing and DSh die is shown in Figure 1. d and e, respectively. The isostrain contours form closed loops around the Centre of the cross-section.

2.2. Optimal reduction for drawing

Considering the problem of choosing optimal reduction, we should take into account that during plastic deformation, all kinds of damage occur and accumulate in the material. Damaging is a quantitative measure of the microfracture of material during deformation. The latter immediately precedes macro-destruction. In accordance with the principle of macroscopic definability, it is believed that the value of damage is uniquely determined by the loading process and is presented as a certain function from this process. The criterion of macroscopic destruction is written as a condition when a measure of damage reaches a critical value.

It is accepted that the increment of damage is proportional to the increment of the degree of shear strain $d\Lambda$ [4] :

$$d\varepsilon = \alpha * d\Lambda , \quad (2.2.1)$$

where α is a proportionality factor determining the intensity of accumulation and development of microcracks.

Moreover, it is assumed [4] that the formation of a macroscopic crack occurs when the damage reaches a critical value ε_{kp} . The degree of shear deformation that the representative volume of the metal has undergone by the time it reaches the ultimate damage is called ductility and denote Λ_p . Assuming that the α parameter is constant for a

given metal, from equation (2.2.1), it becomes $\varepsilon_{kp} = \alpha \Lambda_p$. Dividing the equation (2.2.1) at the last ratio gives:

$$d\psi = \frac{d\Lambda}{\Lambda_p}, \quad (2.2.2)$$

where $d\psi = \frac{d\varepsilon}{\varepsilon_{kp}}$, and ψ is called the degree of exhaustion of the resource of plasticity.

From equation (2.2.2) it follows that:

$$\psi = \int_0^t \frac{Hdt}{\Lambda_p}, \quad (2.2.3)$$

where $Hdt = d\Lambda$ and H is the shear strain rate intensity.

Then, the condition of deformation without fracture becomes:

$$\psi = \int_0^t \frac{Hdt}{\Lambda_p} < 1, \quad (2.2.4)$$

with the condition of destruction:

$$\psi = \int_0^t \frac{Hdt}{\Lambda_p} = 1. \quad (2.2.5)$$

The results of the study of plasticity under proportional loading with sufficient accuracy for practice can be represented by parametric dependences reflecting the relationship of the ultimate degree of shear strain Λ_p with stress indicator $\frac{\sigma}{T}$ (σ is the hydrostatic stress and T the tensile stress intensity).

The most important application of the theory of deformability is not the prediction of the fracture moment according to criterion (2.2.5), but the assessment of metal damage by determining the value ψ . This allows us to predict the quality indicators of workpieces and products associated with damage, and to correctly set up the modes of recovery annealing. To this end, the theory of deformability has developed ideas about the effect of annealing on the value of exhaustion of the plasticity resource [24].

It was noted above that energetically micro-discontinuities can be either unstable or stable. The former can be cured during recovery annealing, the latter cannot. This

circumstance leads to the fact that there are some critical values of ψ^* , above which the annealing efficiency changes. At $\psi < \psi^*$ through annealing, it is possible to completely heal micro-discontinuities and restore the intact metal structure. At $\psi > \psi^*$ only partial repair of the damage is possible. The value ψ^* depends on the metal deformability, its characteristic range of change is $\psi^* = 0.2 \div 0.4$ [4]. There is a second critical value ψ^{**} , after which there is a sharp decrease in the recovery of plasticity resource. When ψ^* is reached untreated micropores begin to appear, then after ψ^{**} (0.5-0.7) [4] untreated microcracks appear.

The simplest version of the deformability theory for calculating the exhaustion of the plasticity resource is the drawing of steel wire. The dependence approximating the experimental curve is:

$$\Lambda_p = \exp(-1.11 \frac{\sigma}{T}) \quad (2.2.7)$$

Where “-1.11” is experimental constant, and when drawing, indicator σ/T varies along the deformation zone and is a first approximation, it can be expressed by the following equation:

$$\sigma/T = 1.73(1 + m \cdot \text{ctg} \alpha) \cdot \ln \frac{f_0}{f} - 1.15 \quad (2.2.8)$$

where m is the friction coefficient, α the half-angle of the die cone, f_0 the wire area before drawing, and f is the current sectional area of the deformation zone.

Equation (2.2.8) shows that value σ/T during drawing is changed along deformation zone from $(\sigma/T)_0 = -1.15$ at the entrance ($f = f_0$), to $(\sigma/T)_1 = 1.73(1 + m \cdot \text{ctg} \alpha) \cdot \ln \frac{f_0}{f_1} - 1.15$ at the exit from the deformation zone ($f = f_1$, where f_1 is the sectional area of the wire after drawing).

The magnitude of the increment of shear deformation during drawing in a first approximation can be specified by the following relation:

$$d\Lambda = -\frac{df}{f} \quad (2.2.9)$$

Substituting relations (2.2.7) - (2.2.9) into expression (2.2.3), the following expression is obtained for the value of the exhaustion of the plasticity resource when passing through the draw:

$$\psi = 0.252 \frac{\lambda^{1.92(1+m \cdot \text{ctg} \alpha)} - 1}{1 + m \cdot \text{ctg} \alpha}, \quad (2.2.10)$$

where $\lambda = \frac{f_0}{f_1}$ the draw ratio

Passing to the single reduction ϕ and taking into account that $\lambda = \frac{1}{1-\phi}$, the exhaustion of plasticity resource can be calculated with the next formula:

$$\psi = 0.252 \frac{\left(\frac{1}{1-\phi} \right)^{1.92(1+m \cdot \text{ctg} \alpha)} - 1}{1 + m \cdot \text{ctg} \alpha} \quad (2.2.11)$$

This equation (2.2.11) can also be used to determine the exhaustion of the plasticity resource in multi-pass drawing.

Using this formula, one can estimate the value of single area reduction, after which it is still possible to restore the damaged metal structure by annealing, and it becomes possible to prescribe annealing rationally. The simple model we developed allows us to study the effect of deformation fractionality on the depletion of the plasticity resource. Suppose you need to get some total reduction ϕ_{Σ} in two passes. The dependence of the total exhaustion of the plasticity resource ψ_{Σ} is independence from partial reduction. The value ψ_{Σ} is estimated by the formula valid for monotonic deformation:

$$\psi_{\Sigma} = \psi_1 + \psi_2 \quad (2.2.12)$$

where ψ_1 and ψ_2 are the exhaustion of the plasticity resource for the first and second transitions, respectively. It is easy to show that:

$$\phi_2 = \frac{\phi_{\Sigma} - \phi_1}{1 - \phi_1} \quad (2.2.13)$$

where ϕ_1 and ϕ_2 are the reductions for the first and second pass, respectively.

Fig. 2 - Dependence of the total exhaustion of the plasticity resource (EPR) [4] on compression during the first pass ($\phi\Sigma=0.4$, $\alpha=8^\circ$, $m=0.05$), calculated by the formula (2.2.11).

Figure 2 shows that redistributing the strain along the transitions can significantly reduce the value of exhaustion of the plasticity resource. Based on the results in (Figure 2) and eq. 2.2.13., at $\phi\Sigma = 0.4$ the most preferred reduction redistribution is: $\phi_1 = 0.23$ and $\phi_2 = 0.22$ (according to eq. 2.2.13). In this case, almost all the damage accumulated by the metal can be healed by means of regenerative annealing ($\psi\Sigma = 0.35 < \psi^*$), while with a single reduction of 40% this cannot be done ($\psi\Sigma = 0.52 > \psi^* = 0.35$).

Thus, during the first reduction, it is more grounded to use 23% of area reduction in the case of conventional drawing. Based on these results, the following scheme of the experiment was chosen (Figure 3). The 1st step is drawing from the round die (5.57mm) to shear die (23% area reduction), while the 2nd step was subdivided into 3 different substeps of classical drawing after shear drawing:

- i - drawing die to 9.3% reduction
- ii - drawing die to 16.6% reduction
- iii- drawing die to 23.2% reduction

Fig. 3 – Scheme of drawing for a model experiment. Percentage shows the reduction degree

2.3. Physical-mechanical properties

The mechanical properties, namely the ultimate tensile strength (UTS) and plasticity (ψ), for the investigated steels were determined from the static tensile test (3 for each measurement) at ambient temperature. To characterize the metal's structural imperfection, the material density, that is a physical property of the material was measured by hydrostatic weighing using the weighing scales AX-200 by Shimadzu. 10 mm-long samples of each diameter were measured six times each. The relative error using this method is 0.003g/cm^3 . Such accuracy allows us to detect micro-imperfections. The maximum change in density, by increasing dislocation and vacancy densities is 0.1% (0.007g/cm^3) [13]. To establish the effect of drawing strain on material strength a Vickers microhardness test was done by applying a load of 200g for 10s. At least 10 measurements were made to obtain an average value.

2.4. Residual stress estimation with SANS

The assessment of the residual stress (RS) state is fundamental to adequately improve knowledge of the effect of intense plastic deformation on metal. Despite the significant amount of data available concerning the constitutive steels of wires, such as those relating to resistance and plasticity, further investigations at an advanced level are necessary for deeper knowledge. The final products, in fact, can present a micro and nano-structural configuration, and a tension distribution still susceptible to improvement. Thus, an

appropriate in-depth investigation aimed at their improvement is essential [25,26]. TKS-400 facility of CANAM NPL infrastructure was used by the Rogante Engineering Office (REO) to analyze the considered wire samples, carrying out RS measurement by neutron diffraction (ND) [27]. The samples were positioned for measurement of the axial and radial components of the strain. Due to the size of the samples, the measurement was carried out only in the center of the wires. The peak positions and their widths were determined by fitting a Gaussian curve to the measured (310) peak of each sample. From the 2θ peak positions, the strains were calculated by using Eq. 2.4.1:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} = \frac{\Delta d_{hkl}}{d_{0,hkl}} = -\cot\theta_{hkl} \Delta\theta_{hkl} \quad (2.4.1)$$

obtained by differentiating d in Eq. 2.4.2 (Bragg law) with respect to θ :

$$2d\sin\theta = \lambda \quad (2.4.2)$$

RS was evaluated from the measured strains by using Eq. 2.4.3.

$$\sigma_x = \frac{E_x}{(1-2\nu)(1+\nu)} [(1-\nu)E_x + \nu(E_y + E_z)] \quad (2.4.3)$$

For stress calculation from the measured strains, it was supposed that in the wire's center the hoop strain is equal to the radial strain. Using this assumption, the radial and axial stresses were calculated using the procedure described in [28].

2.5. Microstructural observations

Microstructural observations were performed using optical microscopy and electron backscatter diffraction (EBSD). As regards to optical microscopy, specimens were mechanically polished following convention (SiC and diamond) and finally chemically etched with a 4% Nital solution. The quantitative estimation of the grain size and its fragments was made in both transversal and longitudinal directions and 100 measurements were made on every micrograph.

For EBSD, the final surface was prepared by electro-polishing in a 65% orthophosphoric acid + 15% sulphuric acid + 6% chromic anhydride + 14% water solution [29]. The important electro-polishing parameters were: temperature 70-90°C, anodic current density 1 A/cm², voltage 23 V and exposure 19s.

EBSD analysis was conducted using a Zeiss SUPRA 55 VP scanning-electron microscope (FEG-SEM) operating at 20 kV. The EBSD data acquisition and analysis were undertaken using the EDAX-TSL Orientation Imaging Microscopy, OIMTM software. The scanned areas were of 100 × 100 μm² with a 0.1 μm step size. To improve the readability of the EBSD data, maps were “cleaned” using standard clean-up options (Grain

dilation/one step and Grain CI standardization). In addition, all points with a confidence index (CI) lower than 0.1 were excluded from the analysis (where CI quantifies the reliability of the indexed pattern). Moreover, to eliminate spurious boundaries caused by orientation noise, a lower-limit boundary-misorientation cut-off of 2° was used. Finally, a 15° criterion was chosen to differentiate low-angle boundaries (LABs) and high-angle boundaries (HABs).

2.6. X-Ray thin structure characterization

To analyze the atomic structures of long-range and mesoscopic orders, diffraction graphs of the samples were captured using the Cr-K α -emission. A V-filtering photographic technique, sensitive to diffusive coherent and incoherent scattering of X-ray beams, was used. This allows analyzing details of structural changes on the atomic order when working with SPD metals [30].

3. Results and discussion

3.1 Physical-mechanical properties of low-carbon steel after drawing with shear.

The first step in the development of continuous severe plastic deformation (CSPD) technique, was the implementation of shear die in the conventional drawing process. As was shown above, the first area reduction was chosen equal to 23%. Such partial reduction provides less accumulation of damage. The present study has shown that at this reduction of die with shear, the strength of wire (UTS) increases from 1210 (initial state) to 1280 MPa (Figure 4). In other words, the die with shear at high reduction degree (23%) results in less strain hardening of the wire, compared to conventional drawing. This is unusual, because often the increase in the reduction degree (compression) must lead to a greater increase in strength [31].

Fig. 4 – Mechanical properties of the wire at the initial state (1) and after a reduction of 23% during the transition from a circle (1) to a shear die (2): left scale – ultimate tensile strength (UTS), right scale – ductility ψ

Furthermore, the plastic properties are increasing in-line with the increase of reduction degree. This is a phenomenon which is not typical in traditional drawing. Indeed, ductility ψ increases significantly with increasing the reduction degree of shear die (Figure 4). This is beneficial for the development of continuous SPD technology, because ductility is the most important item of the manufacturability.

With the aim to demonstrate the behavior of the low-carbon steel properties data for conventional drawing (Figure 4) is shown. Here in conventional drawing the traditional curve evolution of UTS (increase) and density (decrease) is observed and the total EPR is equal to 2.92 if the area reduction at one time is equal to 62.4%. However, using partial reduction with 4 passes it is only accumulated 0.749 total EPR.

Next to shear die, the conventional round die is applied. And here with the purpose of showing an effect of partial area reduction, a set of round dies was used (Figure 3). In this case, a set of the EPR measurements varies from 0.43 (9.3% area reduction on the 2nd pass) to 0.35 (23.2% area reduction on the 2nd pass). It is evidential that based on section 2.2, calculating for the last case is preferable from the point of damage accumulation. The further behavior of the mechanical properties while drawing with shear is shown in Figure

5. As can be seen, with the increase of the reduction degree, the strength properties tend to decrease. Such behavior can be described with the Bauschinger effect and reversed loading at transition from shear to round die leads to making plastic flow easier. However, the results of the mechanical tests do not give us a complete picture of the processes occurring during the drawing with shear.

Fig. 5 Diagrams of property changes in the wire at different degrees of reduction using die with shear ($\varnothing 4.65$ sh –shear die) and round dies: a – UTS; b – Density; 1-initial state, 2 – shear die, 3 – DSh+9.3% reduction, 4 – DSh+16.6% reduction, 5 - 23.3% reduction

Investigations on density measurements showed that while drawing through the die with shear, the density changes in the range of error limits (Figure 5b). In the subsequent reduction, with a round die of 9.3%, wire density increases compared to the density after drawing with shear. Further increase in the reduction when using round dies does not lead to the classical picture of the density decreasing with the increase in the partial reduction. The wire density is also in the range of error limits. An exception is the change of the density after shear dies with a reduction degree of 23%. This means that changing the shear die to the round leads to the healing of the pores [32] by changing the direction of the metal flow. Healing pores improves damage measures and thus increases the plasticity resource. This phenomenon is an additional favorable factor for increasing technological plasticity while drawing with shear. Based on the data density and strength properties, it can be concluded that the combination of the dies with a reduction in 23% yields to the optimal combination of technological properties.

The microhardness behavior also has certain features as well as at first step of DSh and on the second step of conventional drawing (Figure 6). From the homogeneity point, the drawing with shear has less explicit anisotropy of microhardness (Figure 6a). The difference of microhardness between longitudinal and transversal sections is equal to 160 MPa (drawing with shear) versus 204 MPa (initial state). After the first “shear” stage at the cross-section of the wire, the shape of the microhardness distribution looks like a “boat-shape” (Figure 6b). Further conventional drawing leads to flattening this profile. This is also unusual for a pure drawing scheme, as it is commonly known that “V” like distribution of microhardness is attributed to twisting/revolution deformation scheme [33]. The “boat-shaped” microhardness distribution could be an additional proof of the twist-like flow of the metal.

Fig. 6 Microhardness distribution of studied samples: anisotropy of microhardness at shear drawing (a), distribution of microhardness in cross-section (b,c).

3.2 Effect of drawing with shear on residual stress in low-carbon steel wire

In some preliminary ND tests carried out, each wire sample was placed with its longitudinal axis in a vertical position, and a 2 mm wide slit and a hole having a diameter of 3 mm were used respectively for the incident and the diffracted beam [27]. Concerning the successive full ND measurements, figure 8 shows, e.g., the measured (310) peaks for the radial direction, related to two wire samples. Figure 7 a,b report the resulting axial and radial RS for all samples

For shear deformation, the shear produces a large increase of axial RS (about 200 MPa in compression), which significantly decreases after drawing deformations. These subsequent deformations even lead to the appearance of tensile axial RS. Shear deformation also causes some compressive radial RS in the range of 50÷60MPa. After drawing deformation, then, the RS radial component decreases and becomes gradually

tensile (figure7 c,d). Further development of the stresses with increasing drawing deformation has no clear trend. FWHM of the measured ND peaks 310 were also evaluated.

Fig. 7. Measured 310 peaks for the axial and radial direction (a,b). Resulting axial RS (c) and radial RS (d). FWHM of the measured ND peaks (310) for the axial (e) and radial (f). 1-initial state, 2 – shear die, 3 – DSh+9.3% reduction, 4 – DSh+16.6% reduction, 5 – 23.3% reduction

Figure 7 e,f reports the resulting FWHM for the axial and the radial directions for all the investigated samples. The peak width and shape supply information related to imperfections of the crystal structure, i.e., about the grain size and dislocation densities. This analysis qualitatively assessed the evolution of these imperfections with subsequent deformation steps. FWHM increases, as expected, with the intensity of deformation, as shown in Figure 7 e,f. The largest growth occurs during the shear deformation, and the subsequent drawing further gradually adds a number of imperfections (dislocations and/or amount of grain boundaries, due to the grain refinement). ND results have provided substantial data helping to evaluate the effect of shear deformation on RS of low-carbon steels, as well as additional support to complement the information already achieved by using the other characterization methodologies. Knowledge of the RS status can help developing a low-carbon wire drawing technology with needed manufacturability and efficiency, and can play a decisive role also in the debugging of material selection and engineering design requirements [27].

3.3 Effect of drawing with shear on microstructure of low-carbon steel wire

The changes in the grain structure under different schemes of deformation are observed in the wire cross-sections with optical microscopy. Figure 8 shows that when drawing with shear, the microstructure becomes finer: the ferrite grain size decreases and the pearlite colonies are 1.5 times smaller in size. This modification of the microstructure is normal as a large partial area reduction of the DSh must lead to grain refinement. The finest and the most uniform microstructure arises when the DSh is followed with a conventional drawing at 23% area reduction (Figure 8) and both grain refinement and large density of sub-boundaries are observed (Figure 9) by EBSD.

These previous observations are important proof for the following: DSh leads to an intensification in grain growth and then to the sequential grain refinement after the subsequent drawing. Peculiarities of such behavior during DSh with a decrease of strength and an increase in plasticity, is explained by the development of dynamic polygonization. In this case, it yields grain growth due to grain boundary movement and annihilation of micro-voids. In addition, considering grain orientations, a predominance of directions $\langle 110 \rangle$ ($\parallel$ normal direction to the drawing plane) is clearly seen, as indicated by the EBSD orientation distribution maps (Figure 9). For BCC lattice metals, $\{110\}$ crystallographic

planes are most favorable for the dislocation slip. This behavior is conditioned by the deformation scheme.

Fig. 8 Optical microstructure of the low-carbon steel wires a,c – initial; b,d – DSh 23%; e,h – DSh+conventional drawing 9.3%; f,i – DSh+conventional drawing 16.6%; j,g – DSh+conventional drawing 23.2%; a,b,e,f,j – cross section; c,d,h,i,l,g – transverse section.

Fig 9. EBSD characterization of low-carbon steel wires, cross-section. orientation maps a,b,c,d,e,f,j,j,h; grain boundary misorientation – l,g,k,l,m,n,o,p; center –a,b,c,d,l,g,k,l; periphery –e,f,j,h,m,n,o,p; initial –a,e,l,m; DSh+conventional drawing 9.3% - b,f,g,n; DSh+conventional drawing 16.6% - c,j,k,o; DSh+conventional drawing 23.2% - d,h,l,p. IPF, crystallographic direction // DD (Drawing direction)

From the industrial viewpoint, these facts are important because the drawing process of the wire can be continuous due to the absence of breakage due to the healing of pores, which are sources of microcracks formation with subsequent dangerous propagation of the main crack and destruction. These effects allow implementing an increase in the plasticity of the wire without annealing in the drawing process up to a technically necessary degree.

3.4. Effect of drawing with shear on the nanostructure of low-carbon steel wire

Given the peculiarities of different deformation variants on the relation the mechanical properties and nanostructure, it is necessary to consider X-ray data. In the diffraction graph (cross-section and initial state) (Figure 10), intense Debye lines corresponding to the body-centered cubic lattice of the polycrystalline structure are observed. The line from planes (110) has maximum intensity and is thin and symmetric. This indicates a high degree of atomic order for this group of planes. The two other lines from planes (200) and (211) are larger in width. Notably, plane (200) has a flat apex, which means for these families there are subgroups of one-type planes contracted or extended relative to each other. Above the background line, diffusive maximums of weak intensity are identified. This suggests that there are ordered small atomic groups in that strongly disordered deformed structure. Note that there are also some diffusive maximums belonging to the carbide phase $\text{Fe(Mn)}_3\text{C}$. The results point out to the revealing of the multiscale preference of nanostructure of phases occurring in steel: the α -Fe phase (polycrystalline structure) having long-range atomic order and a carbide phase ($\text{Fe(Mn)}_3\text{C}$ nanosized particles) drive into the plastic structure of the matrix, but keeping its crystallographic individuality. Therefore, they reveal themselves above the background in the form of flattened low-intensity maxima, but with clearly distinguishable positions of diffraction angles Θ which are typical for the carbide phase (Figure 10). The Debye maximums of the main α -Fe phase, for DSh sample, have a flat or bifurcate apex, except for the (110) reflection, it has the form of a thin δ -function (Figure 10 f), characterizes the state of planes (110) as the most relaxed and perfect (Figure 10 b). For the other planes ((200) and (211)) of the α -Fe phase, the complex form of their interference apices proves delamination (Figure 10 g) into contracted and extended groups of planes. So, a longitudinal wave effect in the material is registered.

For the consequently drawn samples, the Debye lines of the (200) and (211) plan families, $\Theta > 50^\circ$, the main α -Fe phase have an unusual form. They have the form of group

maximums (wide peak), which means that in the structure of long-range order. There are mesoscopic distortions, including tens and even hundreds of planes of the corresponding type in the form of entire domains inside the material structure. Such locally delaminated states of the mentioned groups of planes are stressed, but they remain in the material and are revealed accompanied by the amorphized fraction from planes (211) with the distorted long-range atomic order. Notably, they have it in the form of nano-sized atomic clusters. The amorphized fraction is in the form of a diffusive halo maximum at the base of the line (211). The Debye line from the family of planes (211) is asymmetric, is split from the side of large angles Θ (Figure 10 d, DSh+conventional drawing 16.6%). They have a bulge with several diffusive maximums at the base (Figure 10 g). At the base of the line, have a diffusive halo band with the maximum corresponding to the angular position of the maximum of the line (211) and pulled in by a decrease in intensity down to line (200). Their flat apex indicates the formation of identical (200) type planes in this group, but with a wide set of interplanar spacings.

The main changes in the diffraction graphs of the sample after DSh combined with conventional drawing 23.2% supplement the described effects for previous samples. The form of the (211) and (200) lines implies the formation of these planes of critical distortions of atomic order with a phase transition (PT) of the second kind of order/disorder type, which is clearly manifested in the form of generating the amorphous fraction in the polycrystalline main structure. This PT led to partial relaxation of elastic stresses in the deformed structure, which is confirmed by the weak intensity of the fluctuations of the diffusive coherent scattering, that have the form of diffuse maximums for the Fe₃C carbide phase over the background line (Figure 10 e).

It is revealed that the effect of spatial delamination of crystallographic planes (211) has on cluster groups differing by a value of interplanar spacing in the range $1.156 < d_0 = 1.172 < 1.178$ Å. The linear size of groups $D = 100\text{--}1100$ Å, number of planes in the group $n = 100\text{--}1000$, and the character of position of the generated groups relative to the unstressed relaxed state (initial): $d_0 = 1.172$ Å, $D = 450$ Å, and $n = 396$. Here, the dispersion of the planes by the value of interplanar spacings was realized in samples (DSh) and (DSh+9.3%), $d = (1.157\text{--}1.177)$ Å, whereas dispersion by the sizes of groups was realized in samples (DSh+16.6%) and (DSh+23.2%), $D = 100\text{--}1100$ Å and $D = 100\text{--}1200$ Å, respectively.

Calculating with X-Ray data, the dislocation density ($\rho_{disl} = \pi \cdot (\frac{\epsilon_{hkl}}{b})^2$, where b – Burgers vector) [34] for hkl (211) after DSh is $\rho_{disl}=13.7 \cdot 10^{13} \text{ cm}^{-2}$ and after following conventional drawing (DSh+Drawing) dislocation density is $\rho_{disl}=6.8 \cdot 10^{13} \text{ cm}^{-2}$ that means two times lower. Thus, softening by deformation is very positive for the development of cost-effective continuous metal forming technologies.

Fig.10 X-Ray spectra for investigated samples of low-carbon steel wires.

During shear deformation in metal, special structures are formed. Owing to “twist” character of the metal flow it becomes possible during further elongation deformation to change the deformation direction. This is very similar to Bauschinger’s effect when the

sign of the load was changed and when the metal flowed in the direction normal to the last deformation step. An additional observed effect of the application of shear deformation is deformation softening [24,35]. Such behavior of metal gives the possibility to continue conventional drawing without any additional treatment. The density of metal influenced by Bauschinger's effect is also increased. This could be indirect evidence of micro voids healing and as a result, increasing plasticity resource. That opens the way to further accumulation of deformation without fracture.

4. Conclusion.

In this work, we demonstrate that the application of the SPD principles generates an effective effect on the low-carbon steel structure. The structural features were considered at several scales, using a wide range of methods. Combining shear deformation with the conventional scheme, the grain growth (reverse grain refinement) process is reached as well as the effect of dislocation annihilation. As a result, the plasticity is enhanced. Residual stress also has a positive influence on manufacturability through the existence of compression stress after shear deformation. These peculiarities of the structure affect the behavior of both mechanical and physical properties (density). The application of the DSh technology leads to a softening effect as well as the healing of micro voids.

These positive effects give the opportunity to increase the effectiveness of drawing technology through controlling plasticity (ductility). It has been shown that in the case of a two-step deformation, there is a parabolic dependence of the EPR measure, and a minimum damage level is reached in a special combination of partial reductions. These results open the way to develop manufacturable continuous technology for steel wire drawing, in particular for welding purposes.

Highlights:

- Shear deformation incorporation into conventional drawing is the advantage. Changing deformation direction in the drawing process gives an opportunity to obtain a higher level of manufacturability.
- For partial reduction in the case of two passes, the first one should be 23%, as that provides an optimum plasticity resource exhausting.

- The advantages of SPD methods were successfully implemented in continuous drawing that gives an opportunity to rationalize or even eliminate intermediate annealing.

5. Author Contributions Statement

A.Z., T.B., M.R. and V.T. performed the measurements, A.Z., T.B., E.P, and H.K. were involved in planning and supervised the work, A.Z., T.B., MR and M.H. processed the experimental data, performed the analysis, drafted the manuscript and designed the figures. A.Z., T.B, F.B. and M.S. manufactured the samples and characterized them with EBSD and SEM. T.B., M.R., M.H., I.K. aided in interpreting the results and worked on the manuscript. A.Z developed theory for partial reduction prediction. A.Z. and H.K. performed FEM modelling with Deform 3D. All authors discussed the results and commented on the manuscript.

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Conflict of interest: Anatoliy Zavdoveev, Thierry Baudin, Elena Pashinska, Hyoung Seop Kim, Francoi Brisset, Mark Heaton, Valeriy Poznyakov, Massimo Rogante, Viktor Tkachenko, Ilya Klochkov, Mykola Skoryk declare that they have no conflict of interest.

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