

Gradient of strength properties in stainless steel wire after cryogenic cooling

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Abstract: This article considers an advanced technology used for processing stainless steel wire, namely cryogenic drawing. The essence of the developed technology lies in the fact that, prior to the beginning of the drawing process, the wire undergoes preliminary heat treatment, which includes quenching at a high temperature reaching 1050 °C. After this stage, conventional drawing is performed, followed by a stage of cryogenic cooling. This process is carried out after each deformation cycle, which makes it possible to significantly modify the properties of the material. A distinctive feature of this study is the use of intermediate heating of the wire to room temperature between deformation cycles. The results of laboratory experiments conducted within this work clearly demonstrate that cryogenic treatment significantly improves the mechanical characteristics of the wire compared with conventional drawing. The application of intermediate heating leads to the formation of a unique gradient microstructure consisting of martensite and austenite. A nanostructured martensitic layer with a grain size of approximately 0.5 μm is formed on the surface of the wire. This grain size gradually increases toward the center of the wire, creating gradient material properties. The research results, presented in the form of detailed tables, graphs showing the dependence of mechanical properties on the number of deformation cycles, and high-resolution micrographs, clearly demonstrate significant differences in the microstructure and properties of the obtained wire.

Keywords: Cryogenic cooling, Nitrogen, Stainless steel, Strength properties, Wire.

1. Introduction

Production of high-strength wire, capable of withstanding extreme loads and maintaining its integrity over a long service life, represents a complex and multifaceted technological challenge [1, 2]. Existing methods, such as multiple-pass drawing, have certain limitations. This process not only leads to a reduction in the wire diameter but also causes a gradual deterioration of the material's plasticity. As a result, the probability of microcrack formation increases, which in turn limits the achievable strength parameters and durability of the final product [3]. Therefore, to obtain wire with truly improved performance characteristics, it is necessary to employ more advanced and modern approaches. One of the most effective solutions to this problem is the application of thermomechanical treatment (TMT). This method makes it possible to effectively control the microstructure of the material at the micro- and nanoscale levels, thereby enabling a significant increase in the strength and wear resistance of the wire [4, 5].

Thermomechanical treatment involves the sequential action of thermal and mechanical factors, which allows not only the improvement of material properties but also the optimization of its internal structure. During TMT, changes occur in grain size and morphology, as well as in the redistribution of dislocations, which ultimately lead to improved mechanical characteristics. For example, such treatment can significantly increase the ultimate tensile strength and improve plastic properties, making the wire more resistant to various external influences [6]. In addition, the use of TMT makes it possible to

substantially increase the service life of the wire, which is an important factor for many industries requiring high reliability and durability of materials. For instance, in construction, the automotive and aerospace industries, as well as in the production of various structural components where wire is used as a primary element, reliability and strength are key factors [7, 8].

To overcome many of the limitations of traditional technologies, increasing attention has recently been given to cryogenic processing technologies. Cryogenic cooling (usually using liquid nitrogen at $-196\text{ }^{\circ}\text{C}$) allows efficient heat removal, suppresses recovery processes, and promotes the accumulation of a high density of dislocations, which leads to significant grain refinement and an increase in strength properties. Studies show that deformation under cryogenic conditions can increase the ultimate tensile strength by 20–40% compared with deformation at room temperature, while maintaining acceptable ductility [9].

However, continuous deformation at extremely low temperatures is associated with several technological challenges, including embrittlement of certain alloys and difficulties in selecting suitable lubricants. In this work, a modified processing cycle is proposed, which includes cryogenic cooling after each drawing pass, followed by intermediate heating of the wire to room temperature before the next deformation cycle. Such an approach makes it possible to combine the advantages of cryogenic structural strengthening with the recovery of ductility and stable friction conditions at each stage [10, 11]. The aim of this study is to analyze the effect of cyclic thermal exposure on the microstructure and mechanical properties of the wire during multiple drawing passes.

In modern wire manufacturing, achieving an ultrafine-grained (UFG) structure is key to producing wire with extremely high strength. Conventional drawing at room temperature (RTD) for materials such as pearlitic steel (e.g., C82D grade) or commercially pure copper (Cu-ETP) is limited by the thermal threshold of the process. At high deformation rates, the temperature in the contact zone can reach 200–400 $^{\circ}\text{C}$, which initiates dynamic recovery and reduces dislocation density.

Continuous cryogenic drawing (Cryo-drawing) involves a process in which both the wire and the die are constantly immersed in a liquid nitrogen environment [9]. This approach suppresses the cross-slip of dislocations, providing a tensile strength approximately 25% higher than that achieved with conventional drawing methods. However, the process also has several disadvantages, including a sharp reduction in ductility and an increased risk of brittle fracture. In addition, lubricants lose their effectiveness at $-196\text{ }^{\circ}\text{C}$, which leads to an increase in the friction coefficient and accelerated tool wear.

Cryogenic cooling after deformation effectively "freezes" the dislocation configurations formed during plastic deformation. According to studies by Volokitina et al. [12], exposure to low temperatures in steels and copper alloys leads to a reduction in the stacking fault energy (SFE), which hinders dislocation cross-slip and climb. As a result, denser dislocation walls are formed, and the fragmentation of the original grains to the nanoscale level occurs more rapidly [12, 13].

In-situ Cryogenic Drawing (continuous drawing in a cryogenic bath) is a process in which the entire operation, including unwinding, drawing through the die, and rewinding, takes place inside a sealed chamber containing liquid nitrogen vapors or splashes. According to Estrin, this approach provides maximum suppression of thermal recovery processes, allowing the highest possible dislocation density to be achieved. However, it may also lead to structural heterogeneity due to lubrication difficulties [14].

Cryogenic pre-cooling involves cooling the billet in liquid nitrogen immediately before it enters the drawing die, while the deformation itself occurs in air. In this case, a sharp temperature increase occurs at the moment of deformation. Studies by Zhang et al. [15] indicate that this creates a gradient structure characterized by strongly refined grains in the center and relatively larger grains near the surface due to rapid heating of the surface layers caused by friction [15].

Cryo-SPD (Severe Plastic Deformation with Cryogenic Holding) is a technology that combines severe plastic deformation methods, such as equal-channel angular pressing (ECAP), with cryogenic holding between deformation cycles. This approach makes it possible to achieve true nanoscale grain sizes ($<50\text{ nm}$) by altering the deformation path. According to Wang et al. [16], cryogenic holding between cycles effectively stabilizes grain boundaries through the interaction with impurity atoms [16].

In this work, a new wire drawing technology is proposed that differs from the approach described in [1, 17]. In a study by Volokitina et al. [17], cryogenic cooling is applied immediately after the wire exits the drawing die. The distinctive feature of the present study is the introduction of intermediate heating to 20–25°C between deformation cycles. Studies by Volokitina et al. [17] demonstrate that such a processing cycle “freezes” the structural defects formed during the previous stage, while returning the material to room temperature before the next deformation step restores sufficient dislocation mobility to prevent wire breakage. This approach makes it possible to achieve higher cumulative degrees of deformation without the need for intermediate annealing.

2. Materials and Methods

The object of the study is a wire made of corrosion-resistant stainless steel AISI 316. To form a homogeneous, equiaxed austenitic structure characterized by high ductility and lower deformation resistance compared to hard δ -ferrite, the samples were subjected to preliminary heat treatment. The preparation regime included austenitization at 1050 °C for 30 minutes, followed by water quenching. This state of the material is considered optimal for ensuring sufficient technological ductility during intensive deformation.

The experimental part of the study was carried out using an industrial drawing machine B-I/550 M (Figure 1). A distinctive feature of the proposed technology is the application of relatively small deformation degrees: instead of the commonly used 10–12%, a range of 5–7% per pass was selected. The reduction of single-pass deformation aims to minimize the gradient of internal stresses and prevent structural defects (such as cracks and fractures), thereby ensuring high uniformity of properties across the wire cross-section.

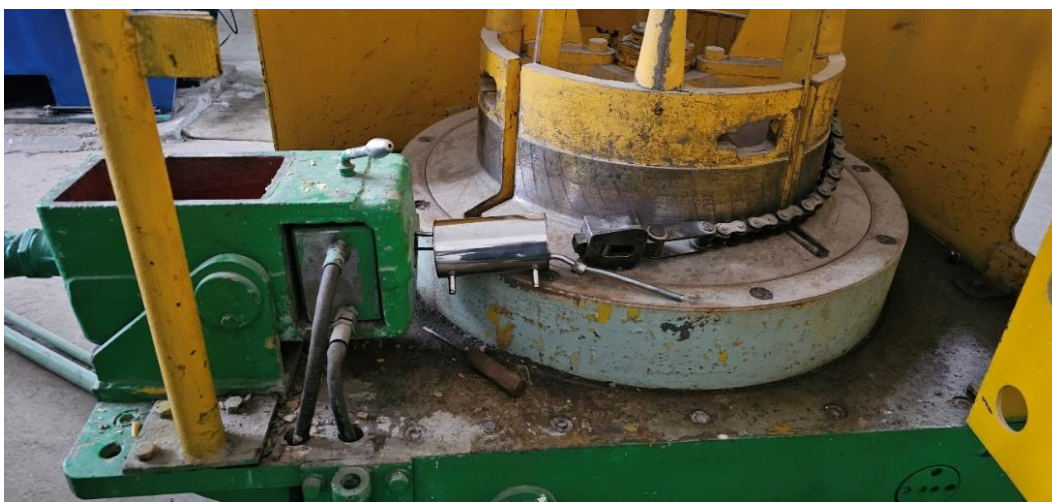


Figure 1.
Installation of the cooling chamber in the drawing machine.

The drawing route consisted of three stages: reduction of the diameter from the initial 6.0 mm to 5.6 mm (individual reduction $\varepsilon = 6.67\%$), followed by a pass from 5.6 mm to 5.3 mm ($\varepsilon = 5.36\%$), and a final pass to a diameter of 5.0 mm ($\varepsilon = 5.66\%$). The implementation of this deformation scheme, combined with temperature control, makes it possible to improve the structural integrity of the wire and the overall efficiency of the process by reducing the probability of deformation-induced damage.

Heating the material to room temperature between deformation cycles acts as a “micro-recovery” stage. This does not cause recrystallization but allows dislocations within fragmented cells to redistribute more uniformly. As a result, the level of local internal stresses decreases, which in purely cryogenic processing methods often leads to the nucleation of microcracks [9]. The main advantage of

deformation with cryogenic cooling combined with intermediate heating lies in the unique control of dislocation evolution. In conventional drawing at RTD (Room Temperature Drawing), dynamic recovery predominates, which annihilates dislocations and limits structural refinement [1].

In this study, a high-precision optical microscope, Leica DM IRM HC, was used to investigate the gradient microstructure of the material. The microscope was equipped with a special attachment for measuring the microhardness of individual phases within the studied sample. This made it possible to obtain detailed information about the mechanical properties of the material at the microlevel. Microhardness measurements were carried out using the Vickers method, which is recognized as a standard of accuracy in this field. The Vickers method is based on the indentation of the sample surface by a diamond pyramidal indenter with precisely defined geometric parameters, specifically an angle of 136° between opposite faces. A controlled load of 0.5 N was applied, ensuring sufficient measurement sensitivity without causing significant damage to the sample.

Transmission electron microscopy (TEM) was also employed, providing extremely high resolution and enabling the investigation of the crystalline structure at the atomic level. This method made it possible to determine the character and density of dislocations, as well as to evaluate the size of subgrains and their orientations.

In parallel with the microhardness analysis, tensile mechanical tests were carried out in accordance with the requirements of GOST 11701-84. The tests were performed at room temperature using a universal testing machine, Instron 5882, which provides high measurement accuracy and controlled testing conditions. The crosshead displacement rate of the machine was set to 2 mm/min, which is a standard value for such tests and ensures reliable experimental results. The obtained microhardness data and the results of the tensile tests were analyzed to achieve a comprehensive understanding of the behavior of the material with a gradient microstructure under various loading conditions. The combination of these methods made it possible to obtain the most complete picture of the structure and properties of the investigated sample.

3. Results and Discussion

During computer simulation of the deformation process using the Deform-3D software package, one of the most important tasks of the theoretical study is the evaluation of the stress–strain state of the material. This evaluation plays a key role because it provides a comprehensive understanding of how the metal changes during processing. Analysis of the deformation state makes it possible to assess the degree of metal working at various points of the billet, which is critically important for understanding the quality of the final product. When studying the deformation state, particularly during the drawing process, special attention is given to the strain intensity parameter, known as equivalent strain. This parameter serves as an indicator of the overall degree of metalworking. Equivalent strain allows one to evaluate the extent to which the metal undergoes changes in shape and internal structure during drawing, which directly affects its mechanical properties. In addition, the study of the stress state during drawing includes determining the conditions under which the level of equivalent stresses remains below the material's ultimate strength. This is particularly important because exceeding this limit may lead to wire fracture in the region located between the drawing die and the pulling mechanism. If the stresses become too high, cracks or even complete wire breakage may occur, which would negatively affect the entire processing operation and the quality of the final product. Thus, the investigation of the stress–strain state during the drawing process not only contributes to understanding the mechanics of deformation but also serves as a basis for optimizing technological processes, ultimately improving the quality and reliability of the manufactured products. Proper control of these parameters is essential for successful metal processing and helps prevent many problems associated with material defects. The Deform-3D software is also equipped with a module for calculating structural transformations during deformation. Using this module, data on structural changes in the deformation zone were obtained, which are presented in Figure 2.

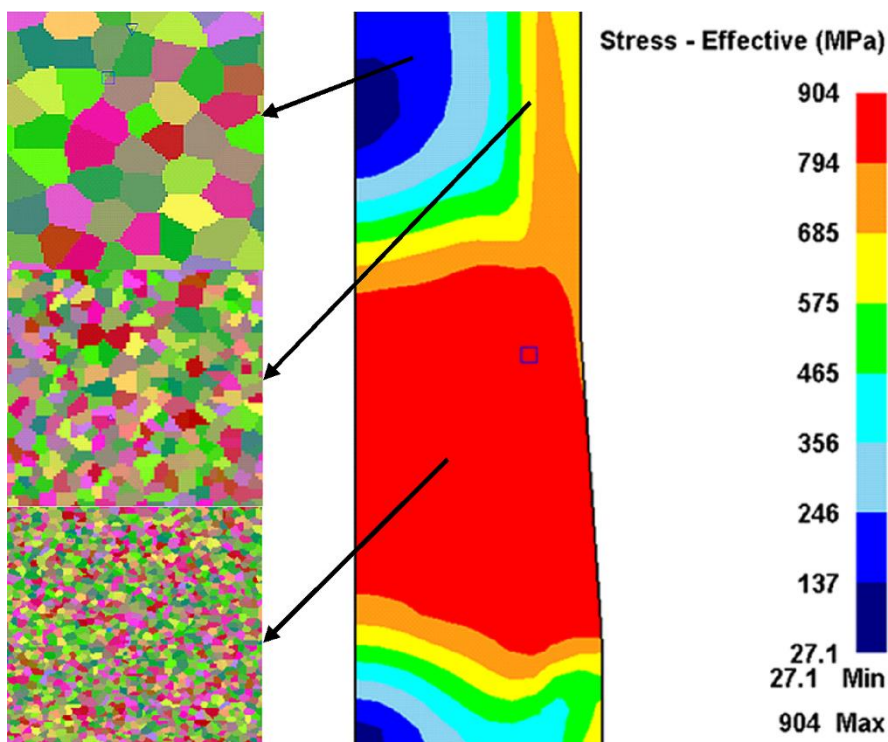


Figure 2. Scheme of structural changes in the localized deformation zone obtained by numerical simulation.

The results of numerical simulation indicate a pronounced gradient character of the formed microstructure across the wire cross-section. It has been established that in the peripheral layers, which are subjected to more intense plastic deformation and higher heat dissipation rates, significant refinement of the grain structure (fragmentation) occurs. In this region, the calculated grain size reaches the submicron level (less than $1\ \mu\text{m}$). At the same time, in the central part of the billet, where the accumulated strain is lower, and the thermal conditions promote a less intensive phase transformation, the average grain size remains at approximately $7\ \mu\text{m}$. The key factor responsible for the formation of such structural heterogeneity is the thermal processing cycle. Intermediate heating to room temperature between deformation stages limits the kinetics of the deformation-induced martensitic transformation. Due to the presence of temperature and stress gradients, the transformation proceeds incompletely and develops unevenly across the cross-section. As a result, a gradient microstructure is formed, characterized by a systematic variation in phase composition and morphology of structural elements from the surface toward the center of the wire. Such a material architecture leads to variability in local mechanical properties, providing a specific combination of high strength and sufficient ductility in the product.

However, the mathematical description of dynamic recrystallization processes and phase transformations at the mesoscale remains complex, which complicates the precise quantitative evaluation of structural parameters. Therefore, it becomes necessary to conduct verification laboratory experiments using high-resolution microscopy methods to confirm the reliability of the theoretical results.

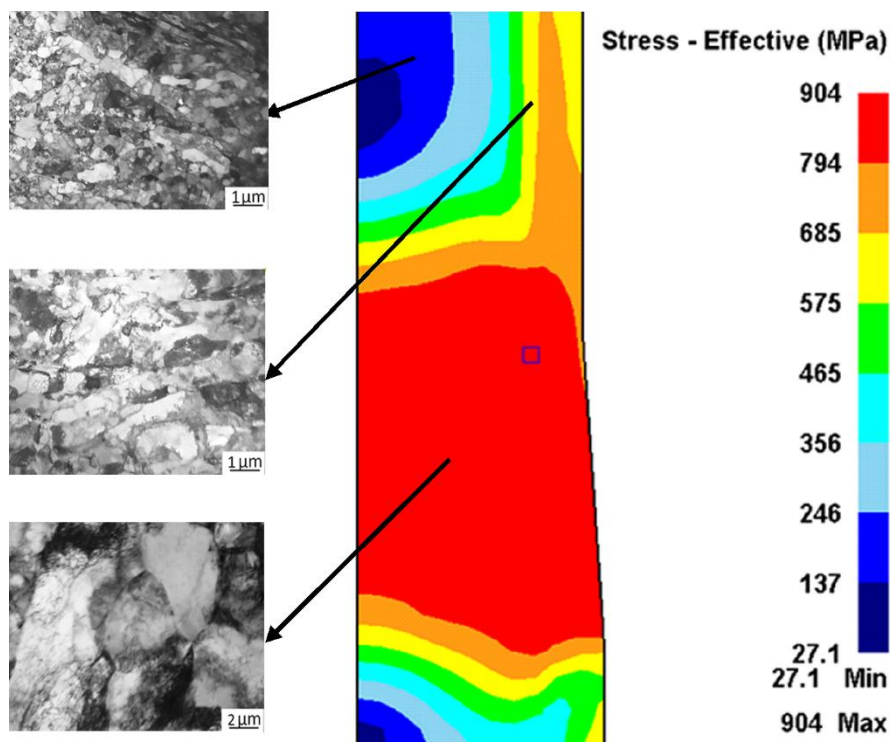


Figure 3.
Microstructure in different regions of the wire.

TEM analysis of the microstructure revealed a pronounced gradient in morphology and phase composition from the axial region toward the surface of the wire. It was established that the size of the structural elements systematically decreases from the center to the periphery: while the grain size in the central zone is about 7 μm , it decreases to 2 μm in the intermediate (neutral) region and reaches minimum values of approximately 0.5 μm in the surface layer. Such a structural topology is caused by the non-uniform distribution of plastic deformation across the cross-section. It is important to note that the intensity of the martensitic transformation also follows a gradient pattern. Nearly complete transformation of the structure into martensite (98%) was observed in the near-surface regions, which are most strongly affected by shear deformation. Toward the interior of the billet, the content of the martensitic phase decreases to 81% in the intermediate zone and 68% in the central region. The obtained experimental data are in full agreement with the results of preliminary modeling, confirming the theoretically predicted patterns.

To verify the symmetry of structural transformations across the wire cross-section, studies of the microhardness distribution were conducted. In the initial state, the material was characterized by a homogeneous distribution of mechanical properties with an average microhardness of 2115 MPa. Analysis of the obtained profiles (Fig. 4) confirms the pronounced gradient nature of wire strengthening. The maximum microhardness values in all deformation cycles are localized in the surface region, which is associated with the highest intensity of shear deformation in these layers. Thus, the average microhardness in the surface zone after the first pass reached 2905 MPa, increasing to 3610 MPa and 3950 MPa after the second and third passes, respectively. The axial region of the wire exhibits less pronounced strengthening, forming a characteristic U-shaped profile of property distribution. Such a topology of microhardness distribution serves as indirect confirmation of the symmetric distribution of deformation-induced martensite across the cross-section of the billet and is consistent with the results of microstructural analysis.

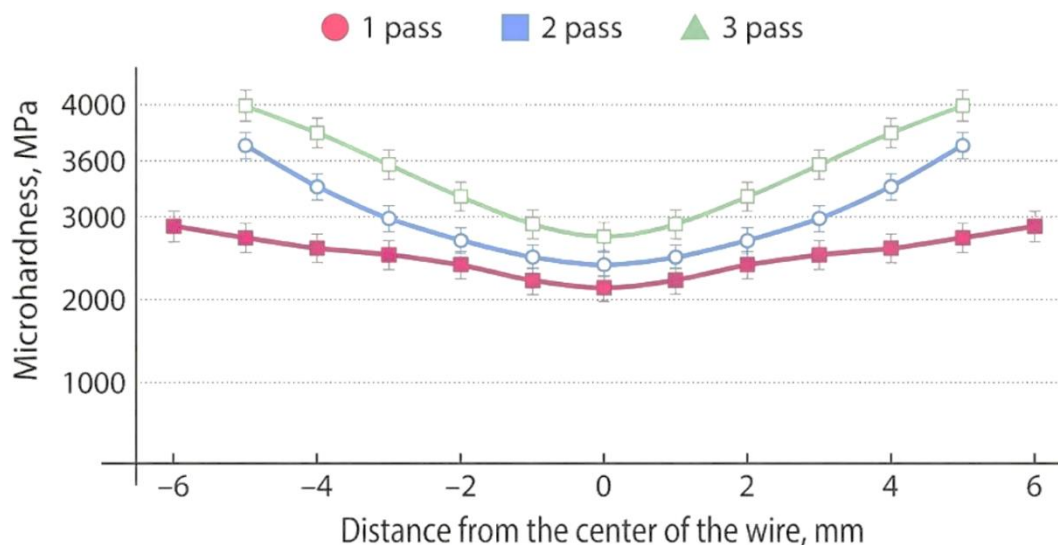


Figure 4. Distribution of microhardness across the wire cross-section after each drawing pass.

The analysis of the mechanical properties of the investigated wire, based on static tensile testing, confirmed the decisive influence of the selected deformation regimes on the combination of strength and plastic characteristics of the material. According to the results of tests performed at room temperature, the steel in the initial quenched (undeformed) state is characterized by moderate strength properties (ultimate tensile strength $\sigma_u = 605$ MPa, yield strength $\sigma_{0.2} = 243$ MPa) and a high reserve of ductility: the elongation $\delta = 40\%$, while the reduction of area $\psi = 52\%$. Such a high ductility reserve of the initial structure makes it possible to achieve significant cumulative degrees of deformation, ensuring intensive strengthening of the material during cold plastic processing.

The application of cryogenic cooling technology in combination with intermediate heating leads to controlled strengthening of the wire. It was found that after deformation using this method, the ultimate tensile strength reaches 910 MPa, while the yield strength increases to 850 MPa. It is important to note that despite the increase in strength characteristics, the ductility parameters decrease only slightly, reaching $\delta = 35\%$, which is significantly higher than the values typically observed in cryogenic processing without intermediate heating [17]. The preservation of a high level of ductile, plastic properties while simultaneously increasing strength is a direct consequence of the formation of a gradient microstructure. The presence of a ductile austenitic core combined with a high-strength martensitic periphery allows the achievement of an optimal balance of mechanical properties, making this technological approach highly effective for the production of high-quality wire.

4. Conclusion

Based on comprehensive studies of the wire deformation process from an initial diameter of 6.0 mm to 5.0 mm in three passes with the application of intermediate heating, the following main conclusions were obtained:

1. TEM confirmed the formation of a pronounced gradient structure across the wire cross-section. It was established that the size of structural elements systematically decreases from the central region to the periphery, ranging from approximately 7 μm to 0.5 μm . In the neutral zone, the grain size stabilizes at about 2 μm , indicating heterogeneity of plastic flow conditions in the metal when inter-pass heating is applied.

2. A significant difference in the intensity of phase transformations across the layers of the billet was identified. The volume fraction of deformation-induced martensite reaches its maximum in the surface

layer (98%), decreasing to 81% in the intermediate region and 68% in the axial zone. This pattern confirms that even with intermediate heating, the main structural transformations remain localized in regions experiencing the highest shear deformation.

3. Microhardness testing demonstrated a significant increase in strength characteristics from the initial value of 2115 MPa to peak values of 3950–3960 MPa in the surface region after the third drawing pass. It was established that the average microhardness in the peripheral zone increases stepwise: 2905 MPa (1st pass) → 3610 MPa (2nd pass) → 3950 MPa (3rd pass). The U-shaped hardness distribution profile across the cross-section confirms the symmetry of the deformation processes occurring in the wire.

4. A high degree of correlation between the experimentally measured microhardness profiles, phase composition, and simulation results confirms the adequacy of the selected theoretical parameters for the deformation process with intermediate heating.

Thus, cryogenic drawing represents a promising technology for processing stainless steel wire, enabling the production of materials with outstanding mechanical properties. The implementation of this technology opens new opportunities for improving the quality and durability of products widely used in various industries, including automotive engineering, aerospace applications, and the production of highly loaded structural components. Overall, the results of this study confirm the effectiveness of cryogenic drawing as a method for enhancing the properties of stainless steel, thereby increasing its competitiveness in the market.

Transparency:

The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

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