ANNOTATION

dissertation work

"DEVELOPMENT AND INVESTIGATION OF A METHOD FOR OBTAINING LONGITUDINALLY NANOSTRUCTURED BLANKS WITH ENHANCED PHYSICAL AND MECHANICAL PROPERTIES",

submitted for the degree of Doctor of Philosophy (PhD)

specialty 6D074000 – "Nanomaterials and Nanotechnologies"

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The purpose of the dissertation work is to develop and investigate methods, deformation tools and devices that combine the most effective types of deformation, such as compression and shear, in a single process. This creates an optimal stress-strain state within the volume of the processed metallic blank, improving its properties and enabling the production of bulk nanostructured metallic materials (BNMM) of sufficient dimensions. Furthermore, these materials have the potential for integration into existing industrial production processes.

Research objectives

In accordance with the aim of the dissertation work, the following objectives were set and achieved:

– an analytical review of available scientific and technical information and the current state of the problem to assess the advantages, disadvantages, concepts, optimal stress and strain modes, and the feasibility of integrating methods of severe plastic deformation (SPD) for BNMM production into industrial processes;

– based on the results of the analytical review, the proposal of concepts for methods that allow for the creation of an optimal stress-strain state within the processed material by combining optimal deformation schemes in a single process. This enables the production of BNMM of sufficient size that can be integrated into existing industrial processes. Additionally, the research provided scientific and theoretical justification for the proposed methods by evaluating their optimality in terms of geometric, stress-strain, and technological parameters using theoretical, computational-analytical, and computerized research methods;

– selection of methods with the greatest potential for industrial implementation and the ability to combine deformation schemes, along with scientific experimentation to substantiate these methods. This involved conducting a series of experiments under various initial conditions, including processing temperature, material composition, crystal lattice structure, deformation mechanisms, grain size, microstructures assessment, physical and mechanical properties of the obtained samples;

– a comprehensive evaluation of the methods based on specific criteria, taking into account all theoretical and practical scientific results, to develop recommendations for their integration into industrial production.

Research methods

When performing the dissertation work, the following research methods were used:

– slip line method for studying the stress state;

– variation calculus method, based on Euler-Lagrange or Lagrangian analysis, for determining displacements and deformation components under conditions of plastic flow;

– finite element method for investigating stress-strain states and energy-force parameters (using the DEFORM-3D software);

– combined solution method for approximate differential equations of equilibrium and plasticity to determine processing efforts;

– light microscopy (LM) for analyzing the initial micro structure of samples (using the Axiovert 200MMAT light microscope);

– transmission electron microscopy (TEM) for analyzing dislocation and grain structures of samples (using the JEOL JEM-1400Plus transmission electron microscope);

– scanning electron microscopy (SEM) for micro structural analysis (using the Tescan VEGA 3 LMH scanning electron microscope);

– electron backscatter diffraction (EBSD) for determining the crystalline orientation of structural elements (Oxford Instruments NordlysMax detector, AZtecHKL software package, HKL Channel-5 Tango software);

– X-ray Diffraction (XRD) for evaluating changes in diffraction peaks and dislocation density using the Williamson-Hall method (using the X-ray diffractometer XPert PRO MPD, Crystallography Open Database (COD), X'Pert HighScore, and Match! software);

– energy dispersive spectroscopy (EDS) for determining the phase composition of samples (using the Oxford Instruments X-MAX80 detector);

– standard tensile and compression testing methods according to ASTM E8 and ASTM E9 for determining strength and plasticity characteristics (using the Instron 5982 tensile testing machine and the WDW-200 universal testing machine);

– Vickers hardness testing method according to GOST R ISO 6507-1-2007 for assessing the hardness of samples (using the Wolpert DigiTestor 930 hardness tester);

– non-destructive eddy current testing method in accordance with GOST 27333-87 for determining changes in electrical conductivity and indirectly assessing thermal conductivity according to the Wiedemann-Franz law (with the eddy current tester VE-27NC).

Main provisions (proven scientific hypotheses and other conclusions that are new knowledge) submitted for defense

– methods based on the combination of deformations optimize the stress-strain state in the metal material and describe it by the slip line fields, stress and strain distribution characteristics, and velocity hodographs;

– methods on the combination of deformations allow to obtain BNMM and it is characterized by TEM and SEM micrographs, EBSD maps, XRD diffractograms, EDS spectra, and histograms of mean grain size distribution;

– results describing changes in micro structure and phase effects in the samples, demonstrating the implementation of strengthening mechanisms such as Hall-Petch

and Orowan, along with results characterizing dislocation-substructure features: TEM and SEM micrographs, EBSD maps, XRD diffractograms, EDS spectra, and histograms of mean grain size distribution;

– metal materials obtained by methods based on the combination of deformations have a high complex of physical and mechanical properties and can be characterized by load-elongation diagrams, load-reduction diagrams, stress-strain (σ–ε) diagrams with an empirical model for mechanical property changes, histograms comparing property values with standards (GOST), Vickers hardness values, specific electrical conductivity, and thermal conductivity.

Description of the main results of the study

As a result of theoretical and experimental research, the following key findings have been established:

– optimal values for the ratio of the diameter (d) to the length (z) of the shear zone and the shear angle (α) have been determined for the implementation of the special forging/drawing/extrusion method ($d/z \approx 1.5 \div 2.0$; $\alpha \approx 20°$) to ensure an optimal stress-strain state within the work piece volume;

– it has been identified for special forging/drawing/extrusion that it is possible to intensify the deformation process by introducing a small shear angle ($\gamma \sim 2 \div 10°$) in addition to the primary shear angle (α). The most rigid deformation occurs during forging without combining it with another process. To intensify deformation during special extrusion, processing should be conducted under counter-pressure. In the case of special drawing with a equal channel die, the cross-sectional area decreases compared to the original section, regardless of the shear angle values. Bending, rather than shearing, occurs, and deformation is not significantly intensified;

– during the process of section shear rolling using a rhomboid-square caliber system, an additional shear deformation with an angle $\gamma \sim 15 \div 20°$ is created due to the distribution of deformations in the cross-section of the square caliber according to the law of point inversion in the subsequent square caliber, in addition to compression deformations;

– SEM analysis of hot-rolled St5ps samples reveals that under the influence of compression and shear deformations, cementite plates within pearlite colonies collide, resulting in the formation of lamellar cementite fragments with sizes ranging from 44.7 to 150 nm. These fragments are not characteristic of the traditional diamond-square calibration system;

– TEM-BF analysis of rolled samples of E110 revealed the formation of micro-shear bands within the structure, with collisions occurring between these micro-bands due to alternating simple shear and compression actions. Dislocation walls also formed in directions approximately perpendicular to the length of the micro-shear bands, which fragmented these micro-bands;

– TEM-BF analysis of samples of St5ps and AD31T1, processed respectively without counter-pressure and with counter-pressure, revealed that in the absence of counter-pressure, less rigid deformation resulted in elongated structures in the St5ps sample, and the grain structure did not develop sufficiently. In contrast, in the AD31T1

samples processed with counter-pressure under cryogenic conditions, equiaxed, well-developed grain structures were formed compared to St5ps.

Substantiation of the novelty and importance of the results obtained

The theoretical results obtained during the investigation of BNMM production methods, particularly stress and strain distribution characteristics, shear angle values, and other geometric and technological parameters, are crucial for optimizing the proposed methods and providing preliminary predictions of micro mechanical parameters. The experimental results, as well as the features, drawbacks, and advantages of the methods identified during the experiments, enable a criteria-based assessment of the production potential of these methods and the development of recommendations for implementation in industrial settings. The proposed methods are oriented towards production and can be implemented in existing metalworking facilities to manufacture metallic products with a high combination of physical-mechanical properties.

Compliance with the directions of scientific development or state programs

The topic of the dissertation corresponds to the priority direction of " Rational use of natural resources, including water resources, geology, processing, new materials and technologies, safe products and structures" of the National Scientific Council under the Government of the Republic of Kazakhstan. Part of the research was also conducted within the framework of grant funding for young scientists as part of the "Zhass Galym" project for 2022-2024 under the above-mentioned priority direction, specifically within project AP14972831, titled "Study of the structure and properties of experimental alumina-matrix composites reinforced with metal carbides and silicon production waste".

Description of the contribution of the doctoral student to the preparation of each publication

The doctoral candidate played a significant role in defining research objectives, planning, conducting both scientific-theoretical and scientific-experimental studies, analyzing, discussing, and summarizing the obtained results, as well as preparing, writing, and submitting printed works either independently or with direct involvement. In most publications related to the dissertation topic, the candidate is the first author or corresponding author.

Regarding the dissertation topic, 25 (twenty five) scientific papers have been published, including: 10 (ten) articles in peer-reviewed scientific journals within the scientific field of the dissertation, indexed in the Science Citation Index Expanded of the Web of Science (Clarivate Analytics) and having CiteScore ratings in the Scopus database (Elsevier), 3 (three) articles in publications recommended by the CQAFSHE MSHE RK, 1 (one) article in a republican scientific journal, 8 (eight) conference paper in the proceedings of international and national scientific-practical conferences, 3 (three) patents of the Republic of Kazakhstan for utility models.