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Gradient Metals' Microstructure Change under Severe Plastic Deformation: A Review

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Research of gradient-structure conditions in metals is rapidly developing area of materials science providing opportunities for creation of materials with unique properties. Long products such as bars and wires are of special interest here as combination of harder surface layer and softer inner part within them allows optimizing a number of mechanical properties. Such gradient shift provides higher durability due to hard outer layer, efficient load distribution across the section, reducing stress concentration in critical points and, more importantly, increasing overall product plasticity. In case of sudden impact, *e.g.*, a hit, softer inner layer may absorb the energy, preventing breakdown. This review study looks into various methods of obtaining gradient metals, mainly based on severe plastic deformation. Their influence on metal-materials' microstructure and properties as well as most efficient areas of application for some of them are studied.

Key words: gradient metals, severe plastic deformation, microstructure.

Дослідження умов ґрадієнтної структури металів є галуззю матеріялознавства, що швидко розвивається та відкриває можливості для створення матеріялів з унікальними властивостями. Особливий інтерес тут представляють довгі вироби, такі як прутки та дріт, оскільки поєднання твердого поверхневого шару та м'якої внутрішньої частини дає змогу оптимізувати

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ряд механічних властивостей. Такий ґрадієнтний зсув забезпечує вищу міцність завдяки твердому зовнішньому шару, ефективному розподілу навантаження по перерізу, зменшенню концентрації напружень у критичних точках і, що більш важливо, підвищенню загальної пластичности виробу. У разі раптового впливу, наприклад удару, м'який внутрішній шар може ввібрати енергію, запобігаючи руйнуванню. У цьому оглядовому дослідженні розглядаються різні методи одержання ґрадієнтних металів, головним чином, на основі сильної пластичної деформації. Вивчається їхній вплив на мікроструктуру та властивості металевих матеріялів, а також найбільш ефективні області застосування деяких з них.

Ключові слова: ґрадієнтні метали, сильна пластична деформація, мікроструктура.

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1. INTRODUCTION

Gradient materials are specific group of materials distinguished by smooth controlled change of chemical composition or microstructure properties by volume. This is not random non-homogeneity but specially created gradient intentionally imbedded in material structure in order to achieve specific properties. Unlike traditional homogeneous materials, whose properties are consistent throughout the volume, gradient materials express gradual shift from one phase or composition to another one. This approach provides massive opportunities for creation of materials with unique properties unachievable with traditional methods [1–3].

The notion of gradient materials was mentioned in international scientific periodicals for the first time in history in 1972, when Massachusetts Institute of Technology published results of a brainstorm by metallurgist M. B. Bever [4, 5]. Although Bever's study was financed by USA's Advanced Research Project Agency, immediate activities did not follow. Only ten years later, gradient metal alloys program in Michigan Technical University in Houghton, MI resulted in a number of publications and reports in conferences. Meanwhile, national program of commercial supersonic space shuttle development started in Japan; thermal barrier coating development for this ambitious project became an objective for an enormous research program and gradient material studies. First results were published only in Japanese, then occasionally in other international periodicals and conferences. The program quickly took off and had impressive results, which were shown in 50 of 70 reports in First International Symposium in Sendai in 1990. At the same time, some laboratories in Germany, Switzerland, France, Belgium, and Finland started independent studies, including ones on gradient material structure.

Gradient-materials' area of use is incredibly wide, including metals, ceramics, polymers, composites and even biomaterials [6–11]. For example, gradient can be used in metal alloys for mechanical strength and durability increase. Take an example of casehardened steel: surface layer is enriched with carbon, which provides high strength and durability, while the inner layer remains plastic and impact viscosity. This smooth transition between hard and soft layer prevents cracking and increases overall reliability of the detail. Similar effect is achieved with steel nitridation, where nitrogen diffuses into surface layer, forming nitride phases increasing corrosion resistance and wearability.

Welding seams, although not always designed as gradient materials, often show gradients of composition and microstructure due to welding process peculiarities. Alloy elements segregation, uneven phase distribution and grain size change are possible, which may significantly influence seams' mechanical properties. Special welding and further treatment technologies are used to minimize these negative effects.

Ceramic materials also can be modified using gradient technology. For example, creation of porosity gradient allows controlling material strength and thermal conductivity. Higher porosity may be necessary in some areas for less weight, lower porosity to provide necessary strength in other areas of component [12].

Material surface hardening using ion exchange or ion implantation is another example of successful gradient technology application. Ion implantation allows implementing alloy element atoms into surface layer of material, creating concentration gradient. This leads to surface physical-mechanical properties change, for example, strength and durability increase [13].

Semi-conductor technologies include multiple examples of gradient structures. Classical p–n-transition in diodes and transistors is basically a gradient structure with smooth change of charge carriers' concentration. More modern semi-conductor devices use much more complex gradient structures to control current and voltage properties [14].

Gradient-materials' application in materials science is caused by inability of most monolithic materials to resist different types of loads simultaneously. This includes mechanical (rupture, compression, bending, fatigue), thermal (high or low temperatures, temperature gradients), chemical (corrosion, oxidation, aggressive environments) and even radiation loads. Engineering objective becomes more complicated considering additional criteria such as environmental safety, raw material availability and cost, reuse and recycle possibilities, and, in case of medical or bioengineering use, biocompatibility.

Gradient materials suggest fundamentally different approach. Instead of rapid transition from one material to another, a smooth transition in properties from core to coating is created [15–17]. This is achieved through controlled change of material chemical composition,

structure or microstructure in the intended direction. This approach allows minimizing inner tensions at the separation line and provides strong cohesion between different zones of the material. As a result, high reliability and longevity of the whole structure are achieved. Various methods are used for gradient-materials' creation: spraying, diffusion sintering, selective laser sintering, *etc*. Method selection depends on specific material and required properties. Gradient transition may deal with not only chemical composition but also microstructure (for example, grain size), which allows to manage material mechanical properties more flexibly. Gradient-materials' study is a promising area of contemporary material science allowing creating highly reliable and durable components for various engineering areas. Further research is concentrated on developing new methods of gradient materials creation, their properties modelling and structure optimization to achieve maximum efficiency in specific areas of application [18–20].

In conclusion, it can be said that gradient materials are promising area in material science bringing new possibilities for creation of materials with unique pre-designed properties outperforming those of traditional materials. Continuous technology development allows creating even more complex and efficient gradient structures, expanding their area of application in different spheres of science and engineering. Research in this area continues, leading the way to new discoveries and innovations.

2. OBTAINING GRADIENT MICROSTRUCTURE THROUGH SEVERE PLASTIC DEFORMATION METHODS

Contemporary technologies allow managing properties' anisotropy intentionally in such processes as drawing and rolling. Change of tense-deformed state may create functional gradients of properties, which is especially important for creation of materials, which should possess various properties depending on terms of use. For example, in aircraft and vehicle industry, it is important to have materials capable of taking high loads in some areas and remain light and flexible in others [21].

Recent years have seen active development of severe plastic deformation (SPD) methods with size reduction of microstructure in specific areas of processed material [22–30]. These methods allow improving materials' strength properties significantly at a sufficient level of plastic deformation. Besides, it should be notes that, with development of nanotechnologies and nanolevel processing methods, possibilities of materials' structure and properties management vary even more. This creates new ways of creating high-functional materials, which can be used in various areas, from medicine to electronics.

SPD technologies have a number of attractive features allowing to obtain materials with ultrafine-grain structure and improved mechan-

ical properties, such as higher strength, hardness, and plasticity [31–35]. At first sight, it seems that SPD should become commonly used, considering its advantages. However, its wider practical use is limited by a number of significant obstacles.

One of main reasons is necessity to apply ultrahigh pressure (5–6 GPa and higher) to achieve desired gradient ultrafine-grain structure. Such pressures create extreme equipment load, causing its faster wearing and, subsequently, higher production costs. Development and application of stronger long-life instruments capable of resisting such loads is one of key objectives for wider SPD use. Research in this area includes development of new instrumental materials, use of innovative coatings and perfection of press equipment structures [36–39].

The second serious barrier is lack of available practice-oriented reference literature describing SPD principles and methods. Lack of detailed manuals and recommendations on technological processes development impairs SPD implementation in industry. Engineers lack information to develop optimal processing modes, selection of proper equipment and product quality control. Effort is required to arrange knowledge on SPD, create learning guides, write review articles and organize special extension courses for engineering and technical personnel.

The third limiting factor is necessity to reconsider all technical-economical concept of production and sales of products obtained using SPD. Despite better properties, ultrafine-grain materials may require changes of existing technological chains, which cause higher expenses. This requires complex analysis of costs and benefits from SPD implementation considering not only equipment and instruments cost but also potential rise of product quality and competitiveness.

Manufacturers' conservatism and unwillingness to give up tried-and-tested technological processes also stops wide use of SPD. Transition to new technology is always connected to risks and requires significant investments. To overcome this obstacle, massive awareness-building effort should be done among manufacturers in showing SPD advantages providing consulting assistance with new technologies implementation.

2.1. Methods for Obtaining Limited Length Billets

There are many ways to obtain gradient microstructure in limited length billets through methods. The most popular method of obtaining gradient microstructure is high-pressure torsion (HPT), where shearing deformation occurs in flat cylindrical samples. In the process sample is placed between two punches and deformed by rotating one punch or rotating both punches in opposite directions (Fig. 1, left) [40, 41].

In the first version, (unlimited) sample may flow radially between punches. Radial deformation here is limited by only internal friction

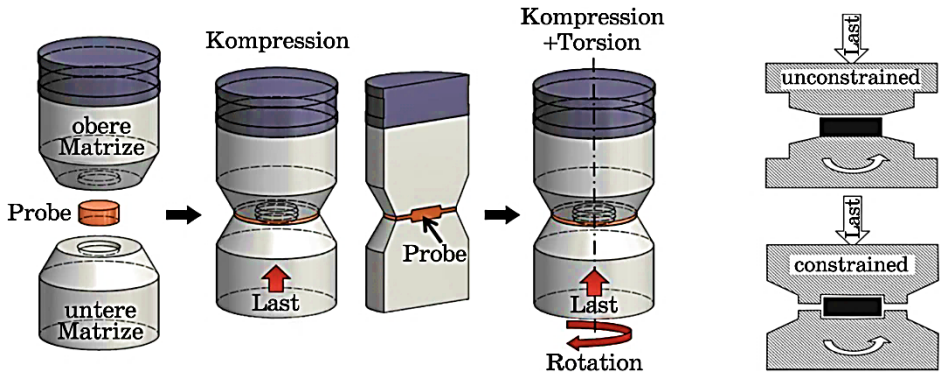


Fig. 1. High-pressure torsion: instruments and operating principle (left, [40]), and difference between 'limited' and 'unlimited' HPT (right, [41]).

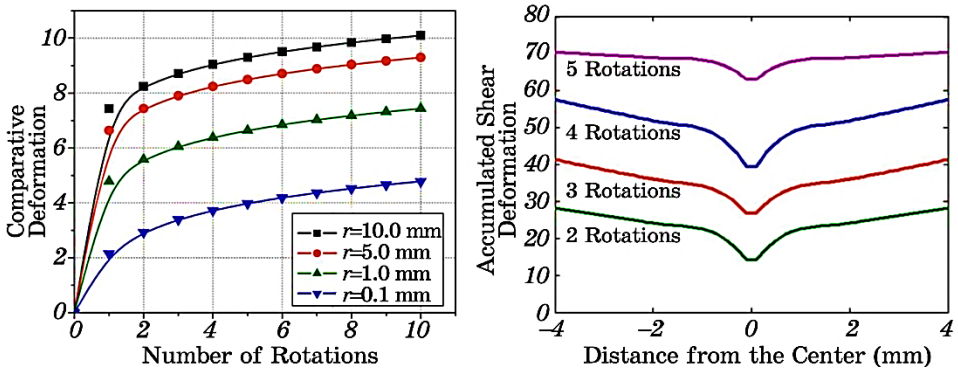


Fig. 2. Comparative deformation (left [41]) and accumulated shear deformation (right [42]) depending on radius and number of rotations.

and friction between the sample and the punch. In this version, sample thickness reduces and diameter increases. Sample thickness in 'limited' version is constant throughout the process. Thus, a distinctive radial deformation gradient occurs depending on number of rotations (Fig. 2).

Accumulated shear deformations rapidly increase radially from disc centre at first, but at longer distances become more linear. Formed microstructures show these gradients in grain size and shape [41].

At the beginning of HPT, as a rule, laminated structures are formed which turn into equiaxial structures as deformation increases due to grain fragmentation, starting from the edge of sample. Average grain size also initially grows from the edge to the centre of the sample. At enough elongation, saturated grain size is reached, when further deformation does not cause structural changes. Thus, highly homogeneous microstructures may be obtained in HPT samples after enough ro-

tations [41, 43].

Another widespread method is equal-channel angular pressing (ECAP). Its idea is the following: a billet is extruded through fitting rig along two channels with the same cross sections crossing at 90–135°. After each pass, the billet is rotated along its axis by 180° (Fig. 3). Deformational impact of ECAP creates various defects of alloy crystals, changing material microstructure. As a result, there is consecutive decrease of grain size due to formation of grid of small-angle boundaries and, then, large-angle boundaries. The advantage is that ECAP can be repeated several times without billet size change, and applied tension can be increased to any level.

Besides, critical shear instability may be caused by deformation direction change, for example, when switching from regular shear (at ECAP) to variable loading with axial compression–extension (at fatigue) (Fig. 4, *b*). Depending on grain boundaries stability in shear

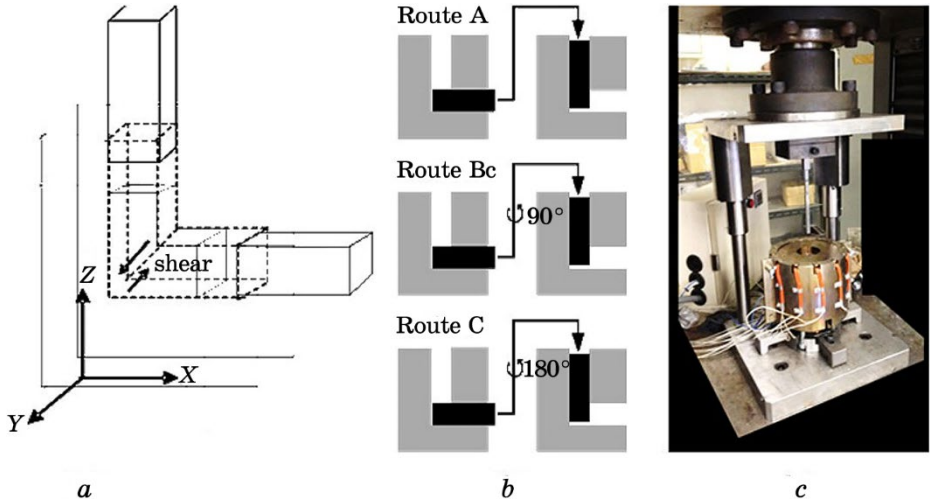


Fig. 3. Equal-channel angular pressing layout [44].

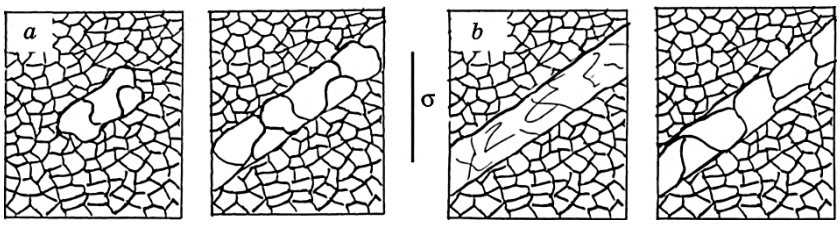


Fig. 4. Alternative models of shear bands formation in UFG materials: local grain coarsening spreading along the shear (*a*); shear band formation through deformation mode change and following coarsening [45] (*b*).

band grain coarsening caused by deformation may occur.

2.2. Methods of Obtaining Long Billets

However, the abovementioned methods practically cannot be used to obtain long billets in industrial conditions. Thus, research of gradient-structure conditions in metal materials is a rapidly developing area of contemporary material science. Long products such as bars and wires, where combination of hard surface layers and soft core provides unique set of mechanical properties, are of special interest. Such architecture allows increasing wearability significantly, evenly distribute loads across bar section and, subsequently, increase overall plasticity and breakdown resistance. This is especially important for details working under high friction or impact loads.

'Conform' method based on SPD principles is used in electrotechnical industry for making heavy-duty copper busbars and cables with higher conductivity [46–48]. Nanostructured implants obtained using SPD are used in medicine and show increased biocompatibility and better acceptability in human body that is promising way for creation of more effective and safer medical products. In addition, there has been research of SPD use in aerospace industry for creation of light and strong materials for aircraft construction as well as automobile manufacturing for creation of details with higher wearability. SPD opportunities are enormous, and further research in this area may lead to creation of new materials with unique properties and expansion of this technology area of application [49, 50].

Main way of metal pressure treatment used in contemporary technological processes of wire making is drawing performed in one-piece die. This process is a key technological operation where two main tasks are solved: obtaining wire with set shape and size and forming required physical-mechanical properties, which define quality and functionality of the final product. For solving the first task size of initial billet (wire rod) should be carefully selected, intermediate billet size should be defined, and drawing transitions for each section should be calculated. This allows optimizing technological process and providing high preciseness of obtained product. Another aspect is choice of proper equipment and tools, which must correspond to set parameters and provide process stability.

The second task is to define steel grade, which will be used in the drawing process and to set optimal values of reduction per pass and overall reduction. These parameters are necessary to achieve set physical-mechanical properties regulated by corresponding state standards and technical conditions. It should be noted that wire properties such as strength, plasticity and corrosion resistance directly depend on choice of steel grade and processing conditions.

However, despite all achievements in drawing, obtaining ultrafine-grain structure providing high physical-mechanical properties of wire using traditional drawing in one-piece die appears to be impossible. This is related to quasi-monotonic character of deformation limiting the ability to achieve required material properties. As a result, researchers and engineers started looking for alternative processing methods that could overcome these limitations.

A classic example of gradient-structure formation is non-monotonic shear deformation during drawing with controlled shear [27, 28]. In case of using low-carbon steel, this method allows to create structure gradient from coarse-grain core to nanostructure surface layers possessing ultrahigh microhardness. Experiments have proven multifold wearability increase for such processed products compared to initial material. However, it should be noted that gradient-structure formation is just one of tools of managing properties. Gradient interaction with other effects such as texturing, phase transformation and chemical composition change opens new possibilities for fine-tuning of local material properties.

Significant potential in this area is shown by dynamic strain ageing (DSA). DSA is seen at severe plastic deformation leading to formation of ultrafine-grain and nanostructural states [51, 52]. Simultaneously with grain size decrease, there is material strength and hardness increase, which is related to point defects and dislocations build-up; this prevents dislocations movement. Combination of gradient-structure formation methods and DSA allows creating products with gradually changing properties from nano- to macrolevel. For example, a product with nanostructural durable surface layer changing into more plastic coarse-grain core, which provides higher impact viscosity.

Managing gradient structure and DSA opens prospects for creation of functional materials with set properties. For example, composite materials where gradient structure improves adhesion between components or a product with controlled cracking resistance, where gradient transitions facilitate crack-propagation energy dissipation. Besides, development of gradient structure methods modelling and computer design allows optimizing technological processes and predicting future material properties. Further research should be aimed at study of various deformation process parameters' (temperature, deformation speed, deformation rate) impact on gradient-structure formation and DSA display as well as development of new control methods and measurement of local properties in gradient-structure materials. In the longer term, this will allow to create high-efficient and durable products for various industries from machinery to aerospace and medicine.

Annealing is used in some cases to restore material plasticity, even if it means less gain in strength characteristics at SPD stage. Depending on specific case, further thermal treatment may simply restore ma-

terial, while another one may cause local recrystallization.

Influence of such annealing is shown in Fig. 5 with types of microstructures and changes in microhardness of non-alloyed copper samples deformed by 8 passes of hot ECAP and then annealed at different temperatures [53].

This tendency shows significant drop of microhardness between 210°C and 250°C. Flow curves between initial copper state and after 8 passes of ECAP show rise of strength properties to the detriment of plasticity, but annealing at 200°C for 20 minutes after SPD shows simultaneous rise of strength and plasticity properties, which is unusual for annealing treatment effect. In authors' opinion, this can be explained by formation of small recrystallized grains with fewer dislocations than in initial grains, but their small size facilitates macroscopic increase of flow stress.

Another promising process allowing obtaining gradient microstructure in bars is cross rolling and one of its types—radial-shear rolling (RSR) patented in Ref. [54]. Its difference from cross rolling is rolling of one-piece bar through three-roll layout with large feeding angles. Unlike well-known processes such as equal-channel angle pressing and Bridgman anvil deformation, RSR has a number of advantages, including lower deformation force, no limits for billet length and ability of rolling with significant elongation ratio without destruction.

Radial shear rolling differs from other metal pressure treatment in the fact that it does not only provide high quality of final product but also allows reducing its production costs significantly. In the process of radial shear rolling, typical spiral macrostructure formation occurs, which is explained by specific force and kinematic conditions appearing during technological tool impact on the billet. Due to this, differ-

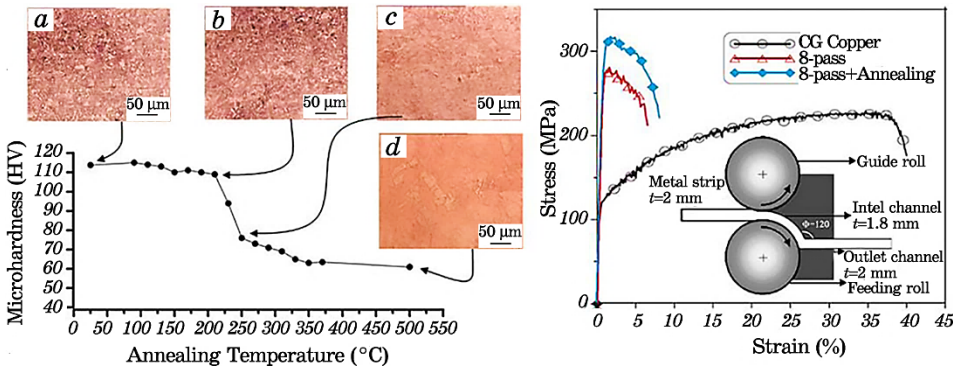


Fig. 5. Influence of annealing temperature on non-alloyed copper after 8 passes of hot ECAP: microphotographs and change of microhardness depending on temperature (*a*) change of flow stress after annealing at 200°C for 20 minutes (*b*) [53].

ent metal layers movement trajectories with different pitch and movement angle of screw lines display complex patterns. As a result, apart from general material structure reduction, there is layer-by-layer orientation of reduced structure, where each layer obtains its own unique movement trajectory, which in its turn influences mechanical properties of obtained product [55, 56].

At RSR, deformation zone follows state of stress close to all-round compression with high shear deformations. In outer layer, each small trajectory-oriented element is exposed to compression deformation along billet radius (diameter), deformation along movement direction and, respectively, stretching deformation across the screwing trajectory (Fig. 6). Metal structural composition elements obtain form of isotropic particles of high dispersion ability separated from each other. Tendency towards formation of structured banded orientation and reticulation caused by general billet elongation is thus suppressed. Such metal deformation conditions do not exist in any known stationary bar-making process. Overall, along the billet volume, metal helicoidal egress within the groove along set trajectories with slowing of surface layers and speed-up of core ones creates volumetric macroshear effect. Macroshear deformations are extremely favourable for non-defect plastic deformation of metal and facilitate significant increase of billets' technological deformability [54].

Authors of the study [58] have shown obtained gradient microstructure of bar section (Fig. 7). However, this method can be used only for deformation of limited length bars, as the authors of Ref. [59] have noted, RSR uses very expensive planetary stands.

Study [60] performed experimental research of RSR technological possibilities for obtaining small-diameter titanium-alloy rods with gradient microstructure from small diameter ingots. Influence of rolling patterns and thermal deformation conditions on macro- and microstructure of obtained ingots and mechanical properties was studied. Production layout, which was experimentally proven at RSR stands,

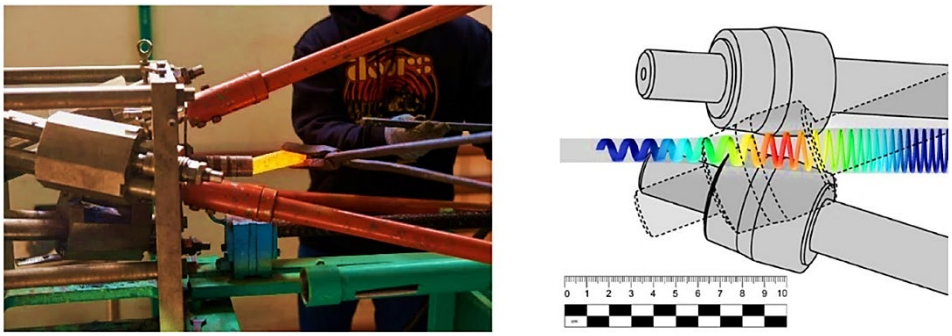


Fig. 6. Radial-shear rolling overview [57].

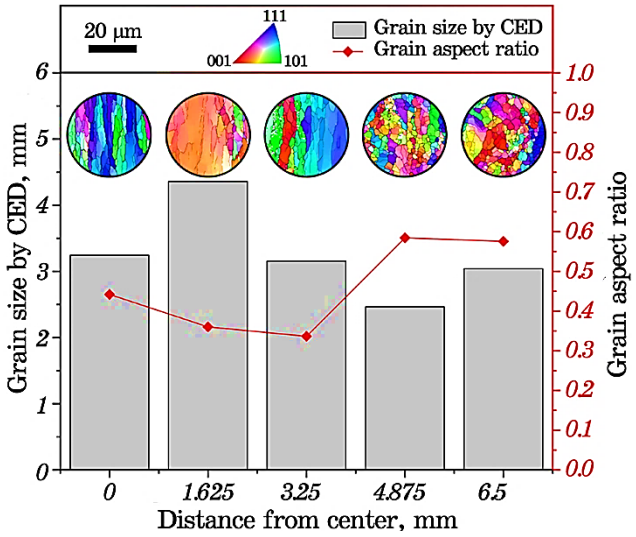


Fig. 7. EBSD study of the gradient microstructures of rolled bar longitudinal sections [58].

can be suggested as foundation for high-technological minirolling line with flexible production plan.

There are studies considering possibilities of combining RSR and drawing [61], since use of traditional drawing as final operation after various SPD processes allows to eliminate all drawbacks (geometrical roughness, pores, *etc.*) of preliminary deformation [15, 26].

Study [26] looks into evolution of copper wires' microstructure and mechanical properties during deformation in rotating equal-channel echelon matrix followed by drawing. As a result of deformation, ultrafine-grain gradient microstructure with high share of large-angle boundary grains was obtained. Use of such strengthened wire in construction will allow reducing structure weight due to smaller diameter.

Study [15] developed new way of wire deforming—'pressing-drawing', which is more effective than previously known ways of obtaining metal with ultrafine-grain structure. The example of tough-pitch copper deformed using pressing–drawing process shows that combination of ECAP and traditional drawing leads to formation of structure with fine homogeneous equiaxial grain with mostly large-angle boundary. These results could be achieved using shear deformations occurring in equal-channel matrix, which caused formation of material structure different from one formed through traditional methods.

As an alternative solution to improve metal deformation process, study [55] suggested innovative combination of radial shear rolling and traditional drawing. This technology forms a unique combination,

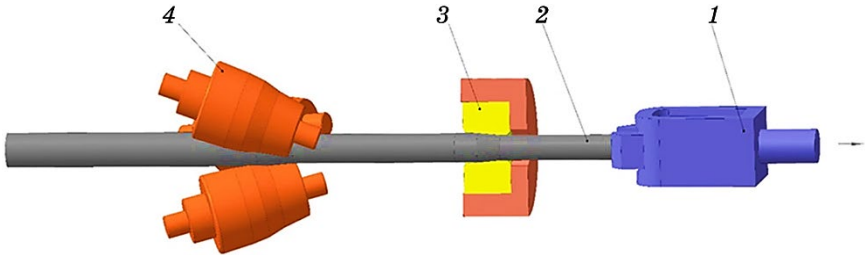


Fig. 8. Rolling and drawing process overview [55].

which allows increasing metal treatment significantly. The process begins from rod rolling through cone-shaped rollers positioned at 120° to each other (Fig. 8). This specific configuration provides even deformation spreading throughout material volume, which, in its turn, promotes structure homogeneity improvement. This is especially important for obtaining high-quality metal products with set properties.

Besides, combination of radial shear rolling and drawing allows reducing overall treatment time significantly. This happens due to the fact that both processes, namely, deformation and rod profile formation, are done simultaneously. This approach speeds up production process and reduces energy and material costs.

During simultaneous rod drawing through rollers and drawing-block there are tension stresses, which, as studies have shown, reduce force required for drawing. This is caused by formation of screw-shaped dents on rod surface after radial shear-rolling step. These dents do not touch drawing-block during drawing, which reduces overall force by 15–17%. This significant force reduction eases the process and increases equipment lifetime [62].

Rod deformation is done using gripping unit, which carefully and effectively provides rod movement through both deforming units. In the gap between these two units, rod torsion occurs that additionally improves mechanical properties of the final product.

As a result of such deformation, gradient microstructure with improved mechanical properties is formed in the rod (Fig. 9).

Thus, suggested solution combines advantages of both radial-shear rolling and traditional drawing, which allows optimizing metal treatment process, improve product quality and reduce costs. Implementation of this technology may bring significant changes in metallurgical industry and open new possibilities for high-quality metal-products' making.

Studies and experience accumulated over the recent years show that radial-shear rolling combines advantage of traditional methods and ability of intense grain-size reduction, even nanoparticles' dispersion and controlled microstructure gradient state.

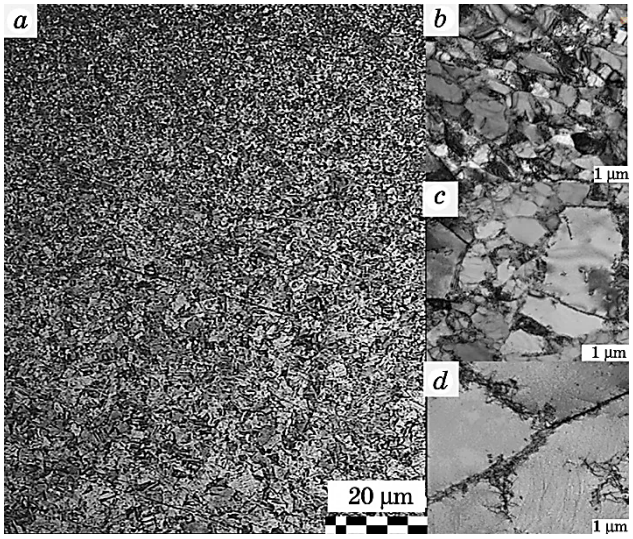


Fig. 9. Rod microstructure after deformation: rod section optical image (*a*); surface area (*b*); neutral area (*c*); central area (*d*) [62].

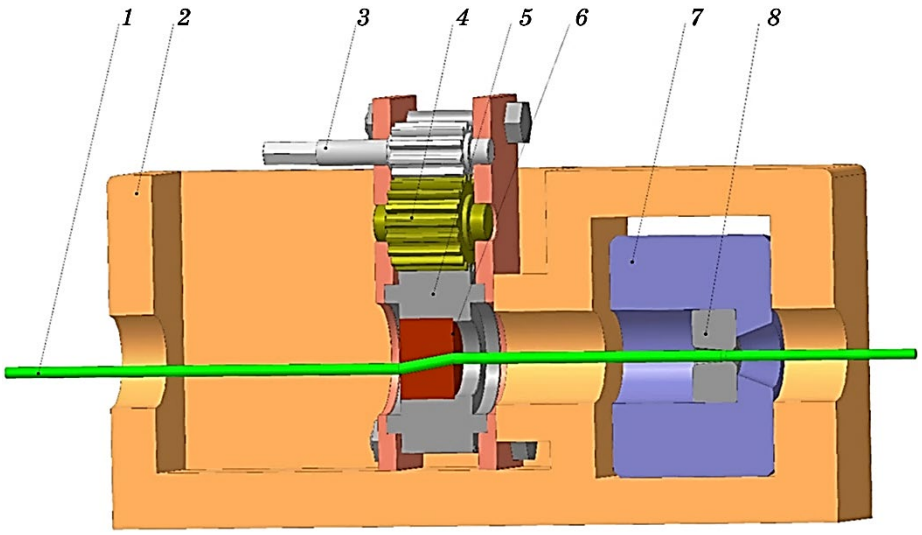


Fig. 10. Wire deformation overview [63].

Research presented in the study [63] looks into evolution of copper wires' microstructure and mechanical properties during deformation in rotating equal-channel echelon matrix followed by drawing (Fig. 10). As a result of performed experiments, ultrafine-grain gradient microstructure with high share of large-angle boundary grains

(Fig. 11) was obtained. This discovery is important for construction industry, as use of such strengthened wire in construction will allow reducing structure weight due to smaller diameter, which in its turn may lead to material saving and better operational characteristics.

Combination of cross rolling and following pressing in angle equal-channel matrix allows to implement stressed–strained state with intense shear of surface metal layers during screw rolling in first deformation zone and combination of intense shear deformation of angular pressing and torsion in second deformation zone. In this case, central part of the billet, which was not fully treated in the first deformation zone, is intensely treated in the second deformation zone through another procedure—in echelon ECA matrix. This creates good conditions for formation of ultrafine-grain structure throughout the whole round billet. Besides, study of angular pressing and torsion parameters is of interest. Technically, this is a process of continuous pressing with billet torsion elements inside the matrix, which increases level of cumulative defor-

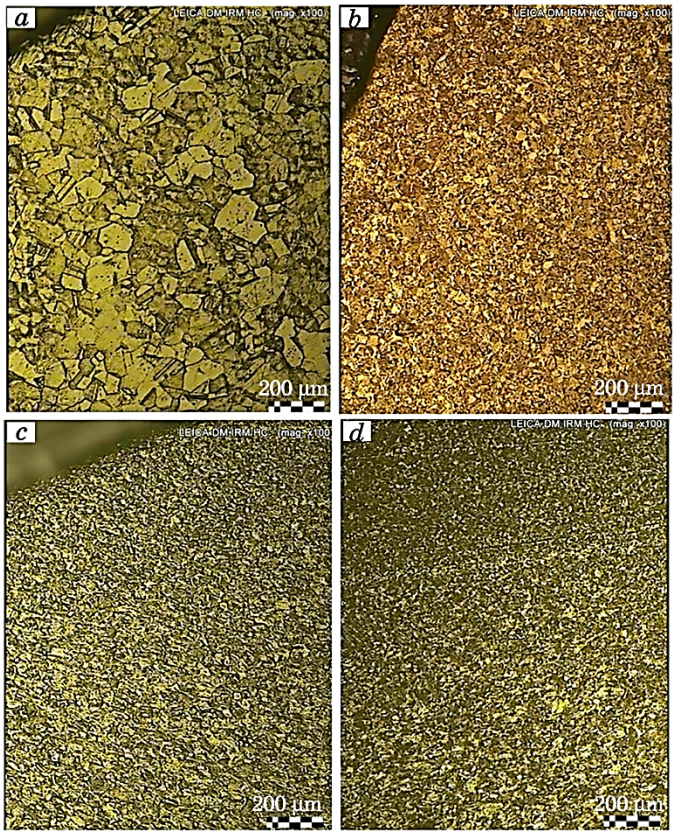


Fig. 11. Copper wire microstructure after three deformation cycles.

mation throughout billet volume and facilitates central-area treatment. Same as in method [38], less homogeneous torsion deformation is covered by more homogeneous and intense shear deformation at ECA pressing, which evens out occurring non-homogeneity after torsion.

Combination of cross rolling and following pressing in angle equal-channel matrix allows to implement stressed–strained state with intense shear of surface metal layers during screw rolling in first deformation zone and combination of intense shear deformation of angular pressing and torsion in second deformation zone. In this case, central part of the billet, which was not fully treated and elongated in the first deformation zone, is intensely treated in the second deformation zone through another procedure—in echelon ECA matrix. This creates good conditions for formation of ultrafine-grain structure throughout the whole round billet.

3. CONCLUSION

Combining gradient structures and thermal treatment brings new possibilities for material properties improvement. Thermal treatment, such as annealing and tempering, may significantly change metal microstructure, which in combination with gradient properties allows achieving optimal combination of strength, plasticity and wearability. This is especially important for components working under heavy loads and temperatures. Thus, analysis of contemporary energy-saving technologies of obtaining metals and alloys with gradient structure and ability of combining them with thermal treatment is an important and promising area of research. This area not only promotes creation of efficient durable materials but also accepts challenges of contemporary industry requiring implementation of innovative and sustainable solutions. In the future, further development of this area can be expected, which will lead to creation of new high-quality metal products with unique properties demanded in various industries.

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