

PHYSICAL FUNDAMENTALS OF THERMOMECHANICAL PROCESSING IN ULTRAFINE-GRAINED METALLIC MATERIALS MANUFACTURING

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Abstract. The processing of metallic materials through the application Thermomechanical Processing (TMP) has now become of major importance in the world research and industrial companies. A great attention is paid to study of mechanism TMP applied to different techniques and technological processes providing ultrafine-grained state of metal materials including submicro – and nanocrystalline ones. The main development in terms of obtaining bulk metallic materials received in the recent years, various schemes of TMP of metallic materials, which allows to realize the severe plastic deformation (SPD). The approach usually propose realization of large plastic strains, providing a well-developed fragmented substructure with the creation of high-angle misorientation between the fragments of the substructure. This paper discusses the physical fundamentals and various methods of thermomechanical processing, applied to single and multiphase steels and alloys ensuring the produce ultrafine-grained (UFG) metallic materials.

Keywords: ultrafine-grained (UFG) metallic materials, severe plastic deformation (SPD), large plastic strain, thermomechanical processing (TMP), fragmented substructure

1. Introduction

The processing of metallic materials with ultra-fine-grained (UFG) structure through the application of severe plastic deformation (SPD) has now become of major importance in many research laboratories around the world [1-7]. Although SPD processing in its modern form including application of Thermomechanical Processing technique is a relatively new development, the fundamental principles of this type of metal processing extend back to the work of artisans in ancient times. A comprehensive review of these earlier developments was presented in [1,2]. In ancient China, during the Han dynasty around 200 B.C. and the Three States dynasty of 280 A.D., the local artisans developed and utilized a new and very effective forging technique for the fabrication of steel for use in swords. The significant feature of this process was that it consisted of a repetitive forging and folding of the metal, which thereby introduced substantial hardening. This repetitive forging and folding process became adopted as a viable technique in the production of high-strength products, and it forms the basis of the famous Bai-Lian steels. Indeed, there is evidence for the use of this procedure in ancient China as early as about 500 B.C. Numerous archeological artifacts are now available from this early period in the form of steel swords and knives, and there are many inscriptions on these ancient objects, which provide a concise record of the processing operation. For

example, a 50-Lian steel sword was prepared using 50 separate smeltings or repetitive forging and folding operations. Subsequently, the processing method spread to Japan and then to India where Wootz steel, a special form of ultrahigh carbon steel, was developed between approximately 300 B.C. and 300 A.D. It is instructive to note that Wootz steel has been specifically designated as an advanced material of the ancient world because of its high impact hardness and superplastic properties at elevated temperatures [8]. Further expansion of this technology to the Middle East led to the development of the famous Damascus steel, which was manufactured in ancient Syria, in the vicinity of Damascus, up to the middle of the eighteenth century when the fabrication technique was lost [9]. However, an important characteristic of all developments in this ancient age is that they lacked scientific rigor, and there was no understanding of the effect of these new processing procedures.

In recent years, great attention is paid to the creation of the physical fundamentals and technological processes providing ultrafine-grained state of metal materials including submicro – and nanocrystalline ones.

It pertains to structural components and to the phases constituting the particular metal or alloy. Advanced approach using the application of severe plastic deformation (SPD) allows to realize the large plastic strains, providing a well-developed fragmented substructure with the creation of high angle misorientation of the boundaries between the fragments of the substructure. The scheme of mesostructure of the grain result in SPD presented in Fig. 1.

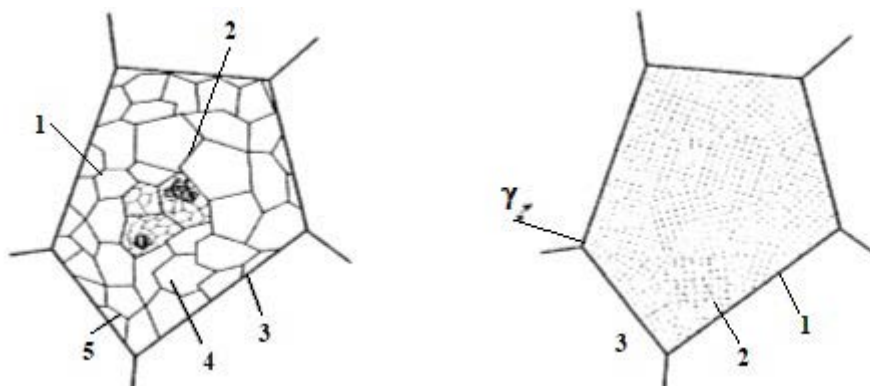


Fig. 1. Scheme of mesostructure of the grain result in SPD: 1 – cell, polygon, fragment; 2 – region of coherent scattering; 3 – grain boundaries; 4 – subgrain; 5 – subboundary; b – misorientation of subgrain volumes; γ – misorientation angle

The second direction in receiving finely divided state is to create technologies that provide a significant refinement of phase as a result of processing. The most effective way of achieving both the above effects applied to bulk metallic materials is Thermomechanical Processing (TMP), which can be used as a standalone technology or in combination of such methods as accumulation roll bonding (ARB) or other similar SPD methods. This paper discusses the fundamentals of TMP, based on the use of hot, warm and cold deformation, in various combinations applied to single and multiphase steels, ensuring the achievement of ultrafine- grained structure with elements of submicro – and nanostructures.

Interest in the processing of ultrafine-grained materials has grown significantly over the last decades [1-4,6,7]. In practice, the presence of a large fraction of high-angle grain boundaries is important in order to achieve highest level of mechanical and functional properties.

The main development in terms of obtaining bulk metallic materials with UFG structural components and phases realized in the last years, various techniques of processing of metals by plastic deformation based on the realization of large plastic strains, providing a well-developed fragmented substructure with the creation of high angle misorientation of the

boundaries between the fragments of the substructure. The second direction to produce UFG structure is receiving a finely divided state by the creation of technologies provided a significant refinement of phase as a result of processing. At the same time alloys with submicrocrystalline (SMC) (0.1 – 1.0 μm) structure manage to be gained with the use of SPD with consequent fixing of initial stages of recrystallization. The base principle of thermomechanical treatment for the production of SMC materials consists in realization at the severe plastic deformations of strongly fragmented structure with the attributes of an amorphous state with a consequent recrystallization. The study of physics of severe plastic deformations indicates, that under these conditions the active role is played with rotation modes of a plastic deformation, result in the formation of strongly misoriented structure [5]. For the realization of SPD the different methods of treatment - shear under pressure, special extrusion and rolling on multiroll mills techniques, all-round forging, ARB etc. can be used [1-4,7,10]. The fragile materials treat at the heightened temperature with its consequent diminution, more plastic - at the room temperature. As the results of investigations testify, the metals and alloys in the SMC condition like nanocrystals have the much changed fundamental parameters – Curie temperature, elastic modulus, diffusion constants [1,7,10]. The SMC materials are conceptually represent a new class of materials with unusual physical and mechanical properties – are anomalous by high strength and damping properties, concerning low temperature superplasticity, heightened viscosity, high magnetic properties [1,7,10]. Though these questions require more extending investigations, but taking into account technological effectiveness of the developed methods of the preparation of SMC structures, it is possible to expect perspectives of their industrial application.

2. Thermomechanical Processing

Last decades it was proved that Thermomechanical Processing is an effective technique to produce metal materials with a nano and submicrostructure elements of the structure [1,7,10,11]. It is known that TMP is one of the advanced resource – saving technologies of metallic stocks and parts of machine production [11-15]. As a result of TMP using rolling, forging, drawing and other metal forming processes it is possible to increase strength and toughness simultaneously applied to the different classes carbon and alloying austenitic and pearlitic steels and alloys, and in the most of cases it will not necessary to conduct heat treatment following by metal forming routinely. The recent ideas of the physics of large plastic deformation had been taken into account for interpretation of structure formation during (TMP) [5,15]. According to advanced ideas the TMP is combination of plastic deformation, heating and cooling (in the different sequence) causing the formation of finished structure are occur in the high density of structural defects (dislocations, disclinations and etc.) conditions induced by plastic deformation.

The main strengthening mechanisms under such type combine influence treatment are - increasing of dislocations density and its more uniform distribution in the volume of metal as compared with annealed condition; forming of dislocation barriers (grains, cells, fragments, subgrains and twin boundaries), the dispersed secondary phases, and etc.; decreasing of the grain size. By now there are many schemes are developed up to days. As a result of TMP realization it is possible to operate by structure and accordingly technological (for example, deformability, machining properties and etc.), mechanical and functional fatigue limit, cyclic durability, corrosion-mechanical strength etc. properties. To the present time the different schemes of TMP – high-temperature (HTMP) and low-temperature (ausforming), preliminary thermomechanical processing (PTMP) and etc. are in detail enough investigated [7,11-15]. The mechanism of regulation of the structure and phase composition with the help of such processing consists in the forming of such dislocation structure, which simultaneously influences and change the phase composition and morphology of the generated phases. It is

known, that the rearrangements of the dislocation structure originating at the deformation in austenitic zone, are a consequence three competing and sequentially of preparing each other processes: a mechanical hardening, dynamic recovery and dynamic recrystallization. In case of single-phase materials in accordance with the lowering temperature at identical deformation parameters there is a changing of the dissipative mechanism. If at the high temperatures of deformation the dynamic recrystallization – most powerful structural mechanism of a dissipation of energy takes place, at the lowering temperature begin to work other weaker dissipative mechanisms. So at the lower temperatures begins to develop fragmentation, and in accordance with lowering temperature there is a fine crushing of the fragments and decreasing of the high angle grain boundaries misorientation. In Figure 2 presented the scheme of typical microstructural elements of UFG structures in metallic materials result in HTMP. At the further lowering of the temperature in dislocation ensemble there is already forms the cellular structure. At the phenomenological level last collective modes of the dislocations movement can be associated with the phenomenon of dynamic recovery. According to contemporary ideas on the plastic deformation of crystals, they show that rotational plasticity modes have arisen in the crystal. A uniformly deformable material cannot further dissipate the mechanical energy supplied to it at a given load rate just by means of plastic shears.

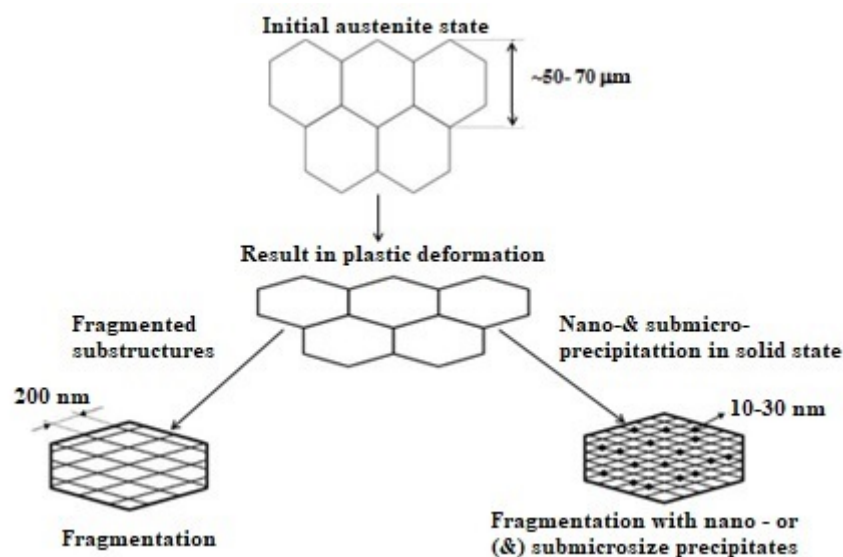


Fig. 2. Typical microstructural elements of UFG structures in metallic materials result in HTMP

The optical properties of the synthesized strontium bismuthate were studied by diffuse reflection spectroscopy (DRS). Diffuse reflectance spectra were recorded in the temperature range 100–503 K. An Agilent Cary 5000 UV spectrophotometer with an integrating sphere was used to record the DRS. BaSO₄ was used as the standard. Also, a special attachment to the spectrophotometer was used, which allows registration of DRS in a vacuum at a controlled temperature. A more detailed description of the design of the device and its capabilities for optical measurements is given in [15].

So it divides into a set of misoriented micro-regions (cells, fragments), each of which starts to swing round plastically during deformation, thereby absorbing additional portions of mechanical energy. As the load rate increases, the rotational modes and their structural indication – fragmentation – will continue to intensify. This continues until the rate of mechanical energy supplied to the specimen exceeds the threshold value, at which a fragmented structure becomes unstable. As soon as that occurs under high temperature

conditions the dynamic recrystallization develops – for a single-phase material the last and most powerful structural mechanism of energy dissipation. In single-phase materials, a drop in deformation temperature results in enhanced strength and reduced ductility and toughness because the dynamic recovery processes are slowed down and more highly stressed structure states characterized by enhanced dislocation density are formed and the presence of strong sources of internal stresses are formed. The latter the increase to plastic shear and the propensity of the metal to the formation of micro-cracks. With these general ideas in mind, it is simple to explain the pattern of structural transformations which we have observed in these experiments dealing with TMP. The marked features of structure formation are confirmed experimentally both on metals, and on steels [7,11-15]. The fine structure and mechanical properties of typical single phase austenitic steel presented in Fig. 3 and Table 1.

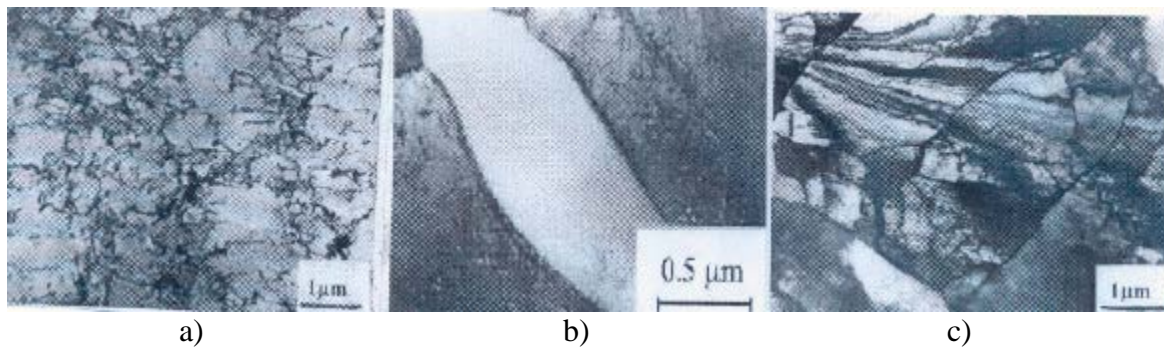


Fig. 3. Weakly misoriented cellular (a), recrystallized (b) and (fragmented (c) structures of 18Cr-10Ni-Ti HTMP treated steel after with single-stage deformation (a, b) and fractional deformation (c): number of passes

a) $n=1$, $\varepsilon = 10\%$; b) $n=1$, $\varepsilon = 50\%$; c) $n=5$, $\varepsilon_i = 10\%$; $\varepsilon_\Sigma = 50\%$

Table 1. Mechanical properties of and structure parameters of HTMP treated corrosion resistant austenitic steel AISI 321 with different numbers of passes n and different rolling reduction, ε

n	ε , %	YS, MPa	Elongation, %	ρ , cm^{-2}	Δr , %	Δf , %	θ , deg.	γ , %
1	10	310	53	1.5×10^{10}	0	10	2.38	0
1	50	245	55	4.1×10^{10}	90	10	5.41	0
5	50	410	46	2.3×10^{10}	0	90	20.39	57

Note: n – number of rolling passes; YS – yield strength; ρ – dislocation density in fragmented structure; Δr – fraction of dynamically recrystallized regions; Δf – volume fraction occupied by fragmented structure; θ – misorientation angle in fragmented structure; γ – fraction of high angle misoriented fragments.

The evaluation of misorientation between fragments was carried out by the technique of [16].

The strengthening kinetics is more complicated in materials with phase transformations result in treatment. The main is inheritance of the deformed structure peculiarities by the resulting structure. First, plastic deformation results in the formation of different structure states in the high- and low-temperature phases, distinguished from one another both by the

temperature peculiarities of dynamic recovery of those phases and by different mechanisms of plastic deformation and work-hardening.

Secondly, after deformation of the high-temperature phase a phase transformation takes place during subsequent cooling which results in additional precipitation hardening of the material (Fig. 4c). The factors mentioned above have different effect on the strength and the ductility. In particular, they may simultaneously result in higher strength and ductility or while raising the strength, they may lower the ductility. So by varying the deformation temperature in the range of the phase transformations it is possible to form structure states which give different combinations of strength and ductility. This is especially important in the use of metal forming treatment for the metals in which the temperature at the end of deformation is lower the phase transformation point.

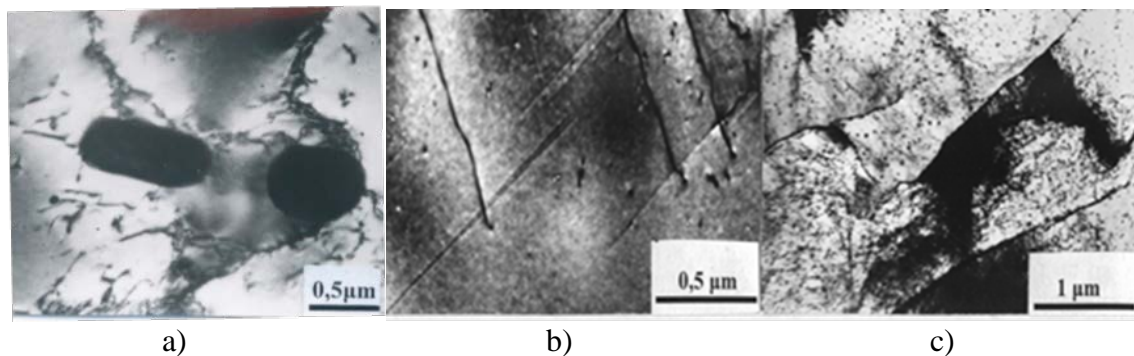


Fig. 4. Fine structure (TEM) of δ ferrite in quenched (a, b) and HTMP treated (c) duplex austenitic-ferritic CrNiTiV steel

A method of multistage pack rolling (MPR), similar to ARB technique, in the TMP regimes has been realized to study effect of TMP to produce UFG structure in ultralow-carbon steel IF type (0.003% C, 0.03% Nb, 0.05% Ti, 0.05% Al, 0.15% Mn) used in the production of automobile steel sheets. A three sheet pack was used as the initial pack for multistage rolling. The sheets were placed one on top of another and rolled at the "warm" deformation temperature: 500-600°C (below recrystallization temperature) with 50% reduction. The obtained band was cut along into halves, one-half was placed on top of the other, and the resulting six-layer pack was again rolled with 50% reduction. The repeated deformation caused welding. The scheme of multipass pack rolling (MPPR) shown in Fig. 5.

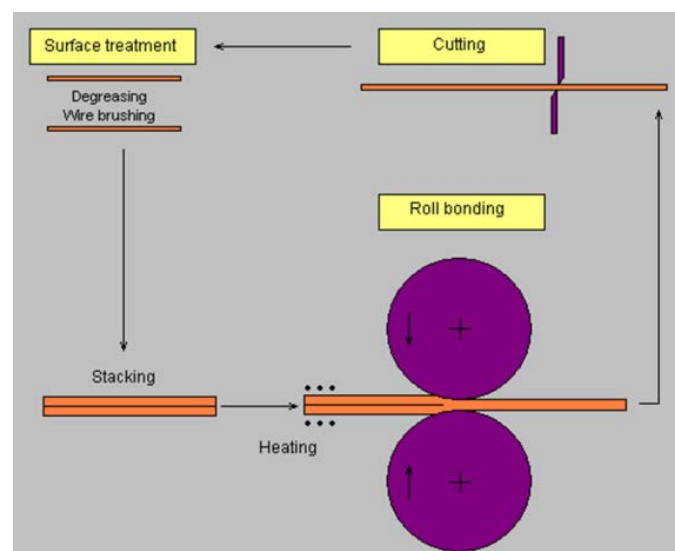


Fig. 5. Scheme of multipass pack rolling (MPPR)

Experimental results showed that "warm" multistage pack rolling produces a submicro-crystalline and subgrain structure with the structural element size of 0.5 - 1 μm in the rolled sheet of ultralow-carbon steel 001 YuT (IF type) (Fig. 6). The obtained structure contributes to raising the yield strength 3.6 times compared to the initial level (after annealing).

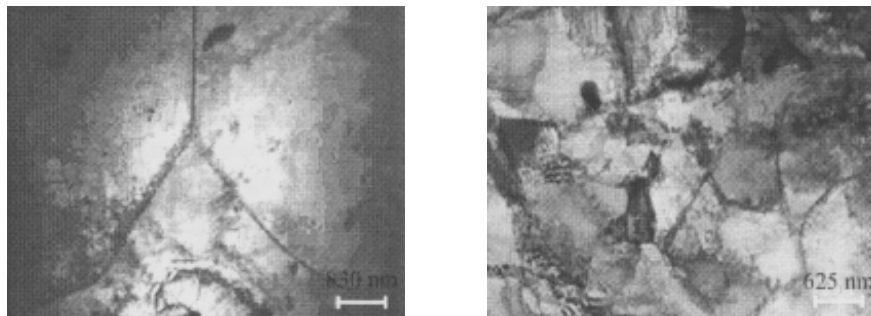


Fig. 6. Fine structure of steel 001 YuT in initial (annealed) state and after pack rolling at 600°C with $\epsilon=1.4$

The numerous experimental data confirm a generality of the above described mechanisms for the steels with the different alloying. The resource-saving technologies TMP-based successfully realized in USA, Japan, Germany, UK, Finland, Russia, and etc. [11-17].

3. Thermomechanical treatment with cyclic phase transformations

The processes of plastic deformation and thermal cycling treatment can be combined. Plastic deformation is always accompanied by the alteration of sensible heat of the workpiece due to the generating heat of plastic deformation, surface friction in the contacting areas as well as heat abstraction to the deformation tool. During the hot rolling of workpieces the temperature of the subsurface layers was observed to change most of all, the sharp reduction of temperature occurring due to the contact with cold rolls. During the pause as a result of the effect of the heat flow from the within of the workpiece the subsurface layers are quickly heated. At the same time, its internal layers, at the moment of passing the deformation area gain temperature and later during the pauses between the passes cool, having warmed up the previously cooled peripheral layers. The temperature drop of the subsurface layers in the deformation area increases with the growth of the compression due to the growth of the deformation area extension and consequently, due to the contact period with the cold rolls. The higher the temperature gradient formed in the deformation area between the workpiece surface and center, the more intensive is further thermal exchange between them. Thus, during the multi-stage rolling it is possible to change the workpiece temperature from one pass to another according to the cycling law by varying the process parameters. As a result in each consequent cycle crystallization will cover both new volumes and those undergone phase transformation before (Fig. 7). Thus, periodic metal deformation in a certain intercritical temperature range induces cyclic partial phase $\alpha \leftrightarrow \gamma$ crystallization in the metal which can be controlled by the selection of temperature-time and deformation rate treatment parameters. The complete course of partial cyclic phase transformations is controlled by co-arrangement of phase composition isolines of metastable system (during heating and cooling) and temperature alteration curve of certain metal volume on temperature-time diagrams of structural state of periodically deformed austenite-ferrite phase system plotted for certain steels. Such approach allowed to produce ultra-fine grained metallic materials [18].

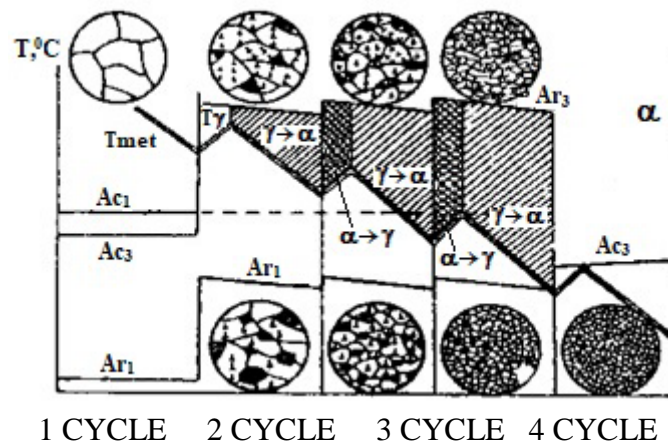


Fig. 7. The scheme of the structure alteration during the $\alpha \rightarrow \gamma$ phase systems periodic deformation [18]

4. Conclusion

- Thermomechanical Processing is an effective technique for production of ultra-fine grained metallic materials.
- The role of mesostructure in Thermomechanical Processing is crucial for obtaining the specified mechanical and functional properties.
- The main mechanisms of Thermomechanical Processing to produce ultrafine-grained structure are the creation dislocation barriers (grains, cells, fragments, subgrains and twin boundaries), the dispersed secondary phases, and etc.; decreasing of the grain size.

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References

- [1] Valiev RZ, Zhilyaev AR, Langdon TG. *Bulk Nanostructured Materials: Fundamentals and Applications*. Hoboken: Wiley/TMS; 2014.
- [2] Wang JT. Historic retrospection and present status of severe plastic deformation in China. *Materials Science Forum*. 2006;503-504: 363-370.
- [3] Valiev RZ, Estrin Y, Horita Z, Langdon TG, Zehetbauer MJ, Zhu YT. Producing bulk ultrafine-grained materials by severe plastic deformation. *JOM*. 2006;58(4): 33-39.
- [4] Langdon TG. Twenty-five years of ultrafine-grained materials: Achieving Exceptional properties through grain refinement. *Acta Materialia*. 2013;61(19): 7035-7059.
- [5] Rybin VV. *Large Plastic Deformation and Fracture of Metals*. Moscow: Metallurgia; 1986. (In Russian)
- [6] Kawasaki M, Jang JI, Ahn BM, Langdon TG. Micro-Mechanical Responses of Ultrafine-Grained Materials Processed through High-Pressure Torsion. *Materials Science Forum*. 2016;879: 42-47.
- [7] Rudskoi AI, Kodzhaspirov GE. *Ultrafine-grained metallic materials*. St. Petersburg: Polytechnical Publishing House; 2015. (In Russian)
- [8] Srivasan S, Ranganathan S. *India's Legendary Wootz Steel: An Advanced Material of Ancient World. Vol. 189-190*. Bangalore: National Institute of Advanced Studies and IISc; 2012. p. 473-481.
- [9] Sherby OD, Wadsworth JJ. Ancient blacksmiths, the Iron Age, Damascus steels, and modern metallurgy. *J. Mater. Proc. Technol*. 2001;117(3): 347.
- [10] Valiev RZ, Aleksandrov IV. *Bulk Nano Structured Metallic Materials*. Moscow: Akademkniga; 2007. (In Russian)

- [11] Jonas JJ, Barnett MR, Hodgson PD. Thermomechanical Processing. In: Groza JR, Shackelford JF, Lavernia EJ, Powers MT (eds.) *Materials Processing Handbook*. CRC Press; 2007. p.1.
- [12] DeArdo AJ. New Challenges in the Thermomechanical Processing of HSLA Steels. *Materials Science Forum*. 2003;426-432: 49-56.
- [13] Kodzhaspirov G, Kim I. *Thermomechanical Processing of steels*. St. Petersburg: Polytechnical Publishing House; 1998.
- [14] Kodzhaspirov G, Rudskoi A. Thermomechanical processing of steels & alloys as an advanced resource saving technique. In: *Conference Proceedings of the METAL 2016 - 25th Anniversary International Conference on Metallurgy and Materials*. 2016. p.248-254.
- [15] Kodzhaspirov GE, Rudskoy AI, Rybin VV. *Physical Fundamentals and Resource-Saving Technologies in the Production of Parts by Plastic Deformation*. St. Petersburg: Nauka; 2007. (In Russian)
- [16] Rybin VV, Voronina YV. Method of orientation matrices in transmission electron microscopy. *Zavodskaya laboratoria*. 1979;12: 1115. (In Russian)
- [17] Kodzhaspirov GE, Karjalainen LP, Speidel MO. Thermomechanical Strengthening of Nitrogen-Bearing Austenitic and Duplex Stainless Steels. *Materials and Manufacturing Processes*. 2004;19(1): 87-94.
- [18] Smagorinskii ME, Gutkin MY, Romanov AE. *Physical mechanisms and technology of steel structure refining in multiple phase transformations*. Leningrad; 1991. (In Russian)