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Research Article

Structure and Mechanical Properties of Titanium Processed by Twist Extrusion and Subsequent Rolling

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The article considers one of the combined methods of severe plastic deformation (SPD), which includes twist extrusion (TE) and subsequent rolling. The use of combined forming methods is promising for industrial use. Titanium grade 1 was used as a material in the experiments. Rolling was carried out in three stages with a decrease in temperature from 350°C to 180°C for a number of passes with one heating. The accumulated strain degree was $e = 4.6$ at twist extrusion and $e = 3$ in rolling. Increasing the reduction per pass decreases the number of heatings and increases the efficiency of the rolling process in whole. At the same time, it is necessary to set the maximum processing modes at which recrystallization processes do not occur in the billet. When rolling, the deformation degree in one pass was taken in the range of 5–20% with an increase in successive passes. The use of such deformation degrees allowed reducing the grain size in titanium grade 1 significantly. Twist extrusion reduces the grain size to 300–500 nm. Subsequent rolling allowed reducing the size of structural elements to 50–100 nm and provided a significant increase in the mechanical characteristics of the billet material (up to 869 MPa) while maintaining satisfactory ductility (up to 11.6%). It was found that increasing the deformation degree in one pass up to 40% at cross-rolling and simultaneously increasing the temperature to 385°C led to a decrease in the UFG structure quality and reduced strength of the deformable material by starting the dynamic recrystallization process.

1. Introduction

It is known that the actively developing technologies based on the methods of severe plastic deformation (SPD) form an ultrafine-grained (UFG) structure in the billet and increase the strength properties of metallic materials significantly [1, 2]. This, in turn, increases the parts' durability and makes it possible to reduce their weight by optimizing their design. SPD processes are actively used as a tool for studying the material behavior both in the process of deformation and after the metal forming processing with the intensification of shear deformations in the billet body or on its surface [3, 4]. Various methods of severe plastic deformation were also applied to study their effect on the structure and grain refinement [5, 6]. In particular, the TE is the SPD method, which reduces the grain size to 100–1000 nm [6]. However, despite the prospects of SPD methods, they are currently of little use in the engineering industry. One reason for this is the limitation on the size of the billets to be machined. The billets obtained by classical SPD methods, such as equal channel angular pressing (ECAP) and high-pressure torsion, have limited dimensions due to the peculiarities of the processes, which reduces the range of products made from processed billets. The second important reason is the need

for subsequent deformation and mechanical and heat treatment of billets. At the same time, it is necessary to apply such processing modes so that neither UFG structure nor properties should be lost. In order to remove the first restriction, new SPD schemes are being developed [3, 7, 8]. To implement them into production, it is necessary to study the structure inheritance and, as a consequence, material properties after exposure to the subsequent deformation and thermal influence. Gaining such knowledge will significantly expand the field of SPD technology application as well as understanding the processes of transformation of constructional material with UFG structure. Therefore, material processing using SPD and traditional metal forming processes are also being actively studied [7, 9–11].

For mass applications, continuous processes of long billet deformation are developed intensively; in particular, different rolling processes are combined with severe plastic deformation [9, 10, 12, 13]. In this case, the material after SPD becomes a semifinished product for subsequent rolling. The reasons for the increased attention to such processes for obtaining materials with a UFG structure are a significant increase in process productivity and the possibility of expanding the range of processed materials. This is an important aspect of the industrial production of parts with high mechanical characteristics [14, 15]. A number of studies point out an additional increase in the strength of parts made of alloys in the UFG state after subsequent plastic deformation. The effect of combining the twist extrusion method of SPD and rolling on the structure and properties of the titanium grade 1 surface layer in the UFG state was studied in [9]. The combined methods are also used to manufacture certain types of parts [16].

The degree of change in the plastic properties of UFG materials after processing depends on the material and the deformation conditions. Most authors observe a decrease in plasticity after SPD [5, 6]. In some cases, a significant decrease in plasticity does not occur due to changes in plastic deformation mechanisms. For example, in [12], it was shown that the use of shear rolling combined with cold drawing allows obtaining wires of small cross sections without intermediate annealing due to the large margin of ductility. However, the depletion of the plasticity reserve of materials with a UFG structure can completely exclude the possibility of further processing [17], especially in processes with tensile stresses at the free surfaces and low hydrostatic pressure.

During SPD, a significant decrease in the size of crystallites occurs. As a result of SPD, the strength characteristics of metals increase significantly with a slight decrease in ductility [18]. Severe plastic deformation leads to the rotation of microvolumes and macrovolumes in the process of metal flow, which explains the anomalous movement of material from the surface into the billet body [4, 5]. Therefore, the ultrafine-grain structure is characterized by the presence of a large proportion of high-angle grain boundaries after billet deformation. The issues of the transformation of the structure of various materials and the texture formation during various types of processing by SPD methods are considered in [19]. At the same time, it is believed that the crystallites formed during the refinement of the initial grains in the process of plastic deformation do not enlarge at a temperature lower than the temperature of the recrystallization beginning [20, 21]. However, grains are refined in the SPD process to a certain size as the processes of return and dynamic recrystallization begins. They prevent further refinement. This process is activated thermally and leads to the cessation of grain refinement due to the rearrangement of the crystal lattice inside and at the boundaries of the crystallites in order to reduce the metal's internal energy [22]. This is especially clearly seen in plastic deformation at low temperatures when the structure resulting at room temperature begins to change without any influence [23]. It is known that rolling at low temperatures leads to strong fragmentation of the grain structure up to a crystallite size of 35 nm [24]. Today, for example, almost all SPD methods of titanium alloys are implemented at temperatures not exceeding 300–400°C to eliminate recrystallization processes in billets. This allows reducing the specific effort in the billet deformation and the load on the process equipment compared to the processing at room temperature.

It can be concluded from this that if the deformation process is carried out at an elevated temperature, then in order to maintain the size of the resulting grain, the temperature should be reduced at the end of the deformation process. To fix the grain size, the billet should be quickly cooled after deformation. This has been studied, for example, when using the multi-axial forging method [8, 10].

Compared to a sufficiently large number of studies on the formation of the material structure in the SPD process, the number of studies on its further transformation during the forming process is very limited. The article [15] notes that copper rolling with the 55% reduction after ECAE reduced the average grain size and its shape and increased the homogeneity of the sample structure. At the same time, the strength and microhardness of the ECAPed sample increase too. In turn, the determination of rational metal forming modes is an important stage in the design of technological processes for shaping complex-profile machine parts. The authors of studies aimed at searching for a range of rational metal forming modes for machining materials with a UFG structure associate them with changes occurring under the influence of force and thermal factors [17, 20]. Thus, one of the main conditions for the use of semifinished products with a UFG structure for manufacturing machine parts is the development of the technology for their subsequent processing by plastic deformation without reducing their strength properties. One of the other most popular forming processes is the rolling process [16].

The continuous structure transformation, which occurs in the process of plastic deformation, is determined by the history of billet deformation. Therefore, the study of the structure formation mechanisms is an important aspect of the development of forming technologies. It is noted in [19] that during deformation by rolling, the structure transforms according to the pure shear mechanism in the central areas of the billet. In the surface layers in contact with the rolls, the

deformation scheme is close to simple shear because of contact friction. The transformation is preserved up to large deformation degrees of about 95%.

To increase the uniformity of properties in the billet volume during SPD, the direction of deformation is changed at each pass. For example, during deformation by the ECAE method with a rotation of 90 degrees, a change in the deformation plane occurs at each pass [15]. In this case, new slip bands appear, the orientation of which differs from those obtained in the previous deformation pass. There is a further crystallite refinement with simultaneous deformation within several grains through which shear bands pass. Similarly, rolling in the longitudinal and then in the transverse direction of the billets must enhance the effect of subgrain refinement. This is due to the appearance of new slip planes. In this case, if we additionally increase the degree of deformation per pass and increase the rolling temperature, then such a change in the deformation parameters can start the dynamic recrystallization process. The pretreatment of the billet by the TE method due to the increase in the accumulated strain and the UFG structure of the billet must also activate the recrystallization processes during the subsequent rolling.

In general, the analysis of papers devoted to factors that allow controlling the SPD process and the UFG structure formation made it possible to identify the main of them. The deformation method and the mode of its implementation have the greatest influence, in particular, changing the direction of deformation routes. A significant influence is also exerted by a decrease in temperature during successive stages of deformation, a change in the degree of deformation per pass, and the total degree of deformation in combined metal-forming processes. It is also important to distribute the accumulated strain between the SPD and metal-forming processes in order to achieve the desired mechanical characteristics of the product. In this case, we can also talk about increasing the economic efficiency of obtaining products by reducing the number of passes during SPD and the subsequent use of conventional metal-forming processes since SPD processes are more labor-intensive. It is also relevant to study the influence of processing modes on the recrystallization process. This will optimize the thermal conditions during the billet forming. In general, knowledge of the processes occurring during combined forming will make it possible to select deformation modes purposefully that provide a given level of product properties.

The analysis of the literature has shown that the issue of the effective application of metal forming methods to billets with a UFG structure has not been studied enough to expand their industrial application. Considering the level of loads and temperatures during the billet processing, dynamic recrystallization can occur. This will greatly reduce the effectiveness of SPD methods in terms of improving material properties. On the other hand, the depletion of the material plasticity during SPD can make its further processing by forming or cutting difficult and not technological. The current lack of unambiguous answers to these questions makes the study of the patterns of changes in the microstructure and mechanical characteristics of billets with a UFG structure during further types of metal forming. Thus, it is of interest to determine the influence of thermomechanical rolling conditions on the microstructure and mechanical characteristics of the rolled strip. In particular, the distribution of the reduction degree and the heating mode between rolling operations will make it possible to determine the rational conditions for obtaining billets with a given ratio of strength and ductility.

The aim of the research was to study the influence of the thermomechanical rolling mode on the mechanical characteristics of the metal and the change in the billet UFG structure, in particular, on the subgrain refinement. This will provide an increase in the efficiency of rolling titanium with a UFG structure and combined forming method in general.

The object of the study was the processes of structure formation in titanium samples from the grade 1 alloy in the combined processing billets by the TE method and subsequent rolling. The subject of the study was the rolling modes after the TE, the structure of the material, and its mechanical characteristics. The influence of the mode of combined plastic deformation on the billet properties was studied in the temperature range of 350°C–180°C.

2. Materials and Methods of Investigation

The combined process of SPD by TE and subsequent rolling was studied. Titanium grade 1, preliminarily deformed by TE, was chosen as the material for the study of rolling mode. The chemical composition of the experimental titanium alloy was determined by the spectral method by using the device "SPECTROMAX" from the company "SPECTRO." The content of gaseous impurities was studied by using an ON900 model gas analyser from ELTRA. It was established that the chemical composition of the experimental titanium alloy is similar to the composition of titanium grade 1 (Table 1).

Deformation by using the TE method was carried out according to the technology [6] on the facility of Donetsk Institute for Physics and Engineering, named after O. O. Galkin (the National Academy of Sciences of Ukraine) with a force of 40 MN, which was modernized with the participation of the authors [25]. The deformation temperature was 400°C; the extrusion velocity was 3 mm/s; the maximum pressure was 1500 MPa; and the back pressure was 500 MPa.

The initial dimensions of the billet section were 39 mm by 24 mm, with a length of 80 mm. The billet was preliminarily processed according to the TE scheme in 4 passes at a temperature of 400°C. It is believed that at this temperature, the recrystallization and recovery processes do not occur in titanium grade 1 [20]. Then, the billets were rolled at the rolling mill 260 in the laboratory of Donbass State Engineering Academy (Kramatorsk, Ukraine). The mill includes a duo working stand with a roll diameter of 260 mm and a barrel length of 200 mm (Figure 1(a)).

One of the tasks of the experiments was to elucidate the influence of the degree of the billet heating on the mechanical characteristics of the rolled samples. The billets were heated in an electric heating furnace SNOL-1.6.2.0.0.8/9-M1 (2.5 kW/900°C). The temperature in the furnace was measured by using a thermocouple, and the temperature of

Table 1: Chemical composition (wt. %) of the experimental titanium alloy.

C	Fe	N	O	H
0.08	0.20	0.04	0.14	0.01

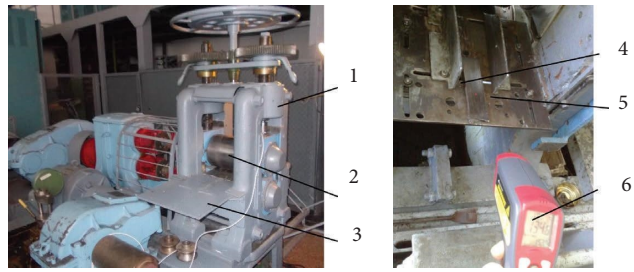


Figure 1: General view of rolling mill 260 (a) and the working stand with wiring (b).

billets was measured by using a hand-held pyrometer with an accuracy of 0.1°C (Figure 1(b)). The furnace temperature during heating was 430°C. The heating temperature of the billets before rolling was 350°C, and in some experiments, it was 320°C and 385°C. This is heating close to the upper limit, at which dynamic recrystallization is guaranteed not to occur in billets with a UFG structure. The heating temperature and time were chosen in such a way as to provide the necessary plasticity on the one hand and not lead to a change in the structure obtained as a result of deformation. In the process of rolling for a number of passes, the temperature was reduced to 200°C. The temperature during rolling was controlled directly at the input wiring and immediately after rolling.

In this work, during rolling, the degree of deformation along the billet thickness was chosen within 95% ($e = 3$). The preliminary four cycles of TE had $e = 4.6$. Partial reductions were taken according to the recommendations of [1] in the range of from 5 to 20%, with an increase in successive passes and 40% in some experiments at the third stage of rolling.

The billet was deformed by rolls with a smooth barrel in several passes. The billet edges (10–15% of their length) are processed significantly less than the middle part during the TE. Thus, the billet central part was preliminarily deformed by TE, and the slightly deformed edge parts were deformed by rolling with the same mode. The microstructure characteristics and properties of the edge parts were used to compare them with the middle part of the billet since it can be considered that they were deformed only by rolling.

The billets deformation by rolling was carried out in 3 stages along the billet axis (Figure 2).

Flat samples were cut out from the obtained billets for stretching and studying their microstructure. The microstructure was studied by using a (TEM) JEM-2100F transmission electron microscope. Tensile tests were carried out on an INSTRON 8801 universal servo-hydraulic machine.

Billets for thin foils TEM observation were cut in the sheet side plane parallel to the rolling direction in the same way as in [26]. The resulting discs and strips 0.12 mm thick were polished in an electrolyte of 30 ml $HClO_4$, 175 ml n-butanol, and 300 ml CH_3OH at a temperature of −20°C. [27]. The characteristic of the surface of destroyed samples under tension [28] was studied on a Superprobe 733 X-ray micro-analyser.

3. Results and Discussion

The billet after 4 TE passes is shown in Figure 3(a). The sample dimensions in the cross section remained practically unchanged, and the length increased due to a change in the shape of the workpiece ends. The change in the workpiece shape shows that it is necessary to continue work on the choice of lubricant and material for the false workpiece for the industrial implementation of the TE process.

The study under a TEM has shown that the microstructure of titanium after TE is represented by a set of strongly off-oriented grains (Figure 3(b)). Dislocation clusters are absent in the bulk of most grains. The pattern of microdiffraction presented in Figure 3(b) is characterized by a significant number of point reflections located over a ring, which indicates the formation of a granular structure with large-angle off-orientation of interfaces. The azimuthal smearing of reflections also shows a considerable level of internal stress. It can be seen from Figure 3(b) that the grain sizes are somewhat inhomogeneous, which substantially influences the mechanical properties of the titanium [29]. The major part of the grains (about 84%) has sizes ranging from 40 to 400 nm. This proves the formation of a submicrocrystalline structure by TE [29].

During the rolling, the temperature and deformation modes of the billets at each stage and pass changed depending on the current geometry of the billets, in accordance with the permissible degrees of plastic deformation and the equipment capabilities.

The heating temperature of the incoming billets before rolling was 320°C or 350°C (Figure 4). The accumulated logarithmic degree of deformation during rolling by stages was 0.91, 2.2, and 2.92, respectively. Depending on the ratio

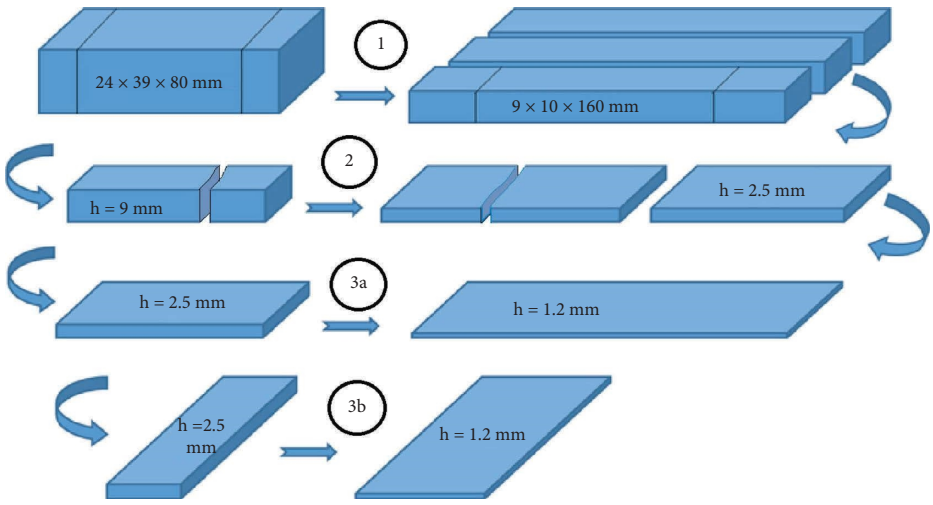
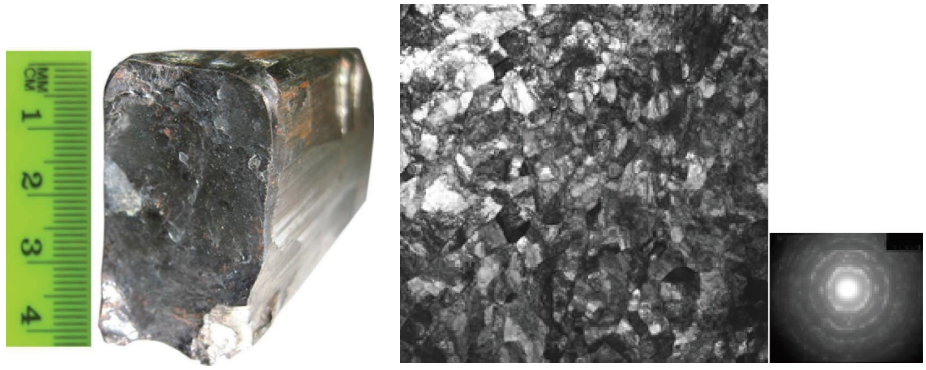


Figure 2: The scheme of the stages of rolling samples after TE: stages 1, 2, 3a indicate lengthwise rolling and 3b indicates cross rolling.



(a) (b)

Figure 3: General view (a) and microstructure (b) of a billet after four passes of processing with TE. Billet dimensions after TE: billet section is 39 × 24 mm; length is 80 mm.

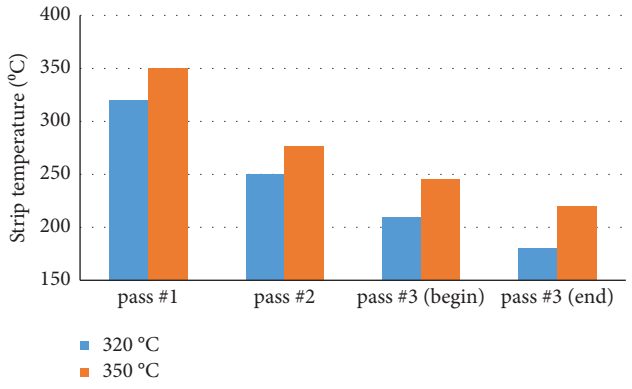


Figure 4: The change in the billet temperature at the first stage of rolling after TE. The temperature of the billets before rolling was 320°C or 350°C. Rolling occurs in 3 passes from one heating.

of the billet dimensions at the processing stages, up to 3 passes were carried out without preheating the billet so that the temperature of the end of rolling was within 200°C. The change in the temperature of the billets at the passes for the first stage is shown in Figure 4. Performing several passes with one heating allows fixing the resulting grain. The fractional deformation, at the same time, eliminates the effect of heating from plastic deformation on the billet structure.

It can be seen from Figure 4 that the temperature decreased unevenly in the passes. Decreasing the rolling start temperature to 320°C lowers the temperatures in all passes. The nature of the temperature change in the passes does not change.

In the first stage, with large thicknesses of the incoming billet (thickness values were in the range of 24.0 to 9.7 mm), deformation was carried out in 4 passes. After one heating, rolling was performed in groups of 3 passes, as shown in Figure 2. After 3 reductions before heating, the billet geometry was controlled. The billet thickness after the first stage of deformation was 9.7 mm; its length was 175 mm. The degree of accumulated logarithmic deformation at the first stage of deformation by rolling was $e1 = 0.91$. The sample shape after TE and the first stage of rolling is shown in Figure 5. The figure shows the deformed edges of the workpiece. There was slight delamination of the metal on the side surfaces of the workpiece; no cracks were observed.

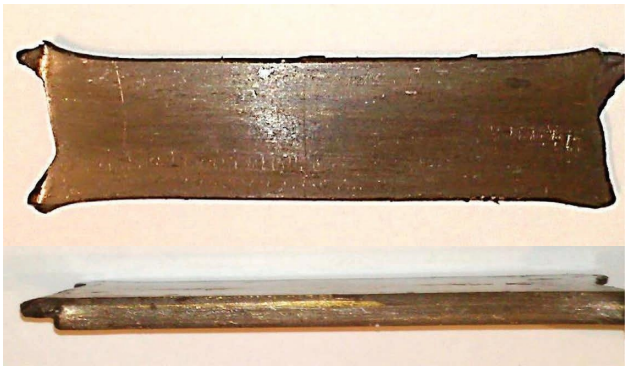


Figure 5: The sample shape after the TE and after the first stage of subsequent billet rolling. Sample dimensions after deformation: thickness is 9.7 mm, width is 44 mm, and length is 175 mm.

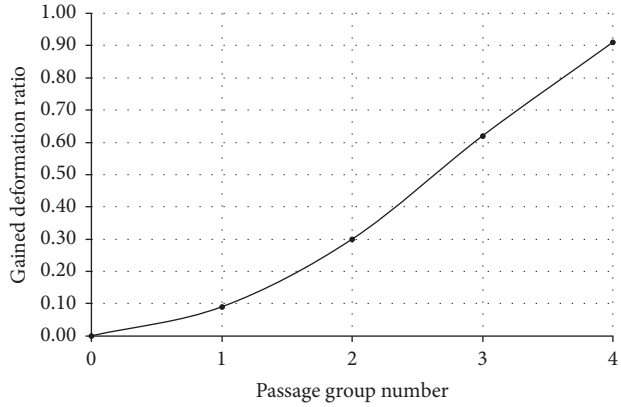


Figure 6: The change of the accumulated logarithmic degree of the deformation at the first stage of billet rolling after TE in passage groups: temperature range of rolling is 350°C–200°C; there are 3 reductions in 1 passing after 1 heating; change in billet thickness during rolling is 24–9.7 mm.

Reductions in each pass averaged 1.2 mm. The value of reduction per pass is taken constant in order to increase the degree of deformation per pass as the billet thickness changes. This made it possible to determine the effect of the accumulated deformation degree on the microstructure and mechanical characteristics of the billets. The change of the accumulated logarithmic deformation by passage groups at the first stage of deformation is shown in Figure 6.

At each stage of rolling, sections were selected from the billets and corresponding thin sections and test samples were made from them. Microstructure analysis was carried out by TEM to compare two parts of the billet. The microstructure after the first stage of rolling the billet with a thickness of $h = 9.7$ mm from the edge part of the billet (without complete processing by the TE method, Figure 7(a)) and the central part of the billet after the TE and rolling (Figure 7(b)) makes it possible to reveal the effect of TE.

As it has already been noted, the edge parts of the samples deformed by TE are not processed to the full extent and can be used to compare the results of processing the material by the combined TE and rolling method (Figure 7(a)) and the material that was only rolled (without complete processing by TE) (Figure 7(b)). The grain size is $n * 10\,\mu$m. The figures show a coarse-grained structure with a high density of dislocations and point defects. After TE and rolling, cells with low-angle boundaries were formed in the material (Figure 7(a)). Figure 7(b) shows the coarse-grained defective structure of the sample after rolling. Arcuate strands on microdiffraction indicate a smoothly changing misorientation inside the grains and the absence of low-angle boundaries, i.e., the billet edges are less deformed than the middle part of the billet.

After rolling, the billet surface was processed and cut lengthwise into three parts with a cross section of 9.0 mm by 10.5 mm and a length of 175 mm (Figure 8). At the second stage, each part of the billet was deformed separately (Figure 2, stage 2) in 6 passes with the following deformation degrees: 4 passes of 7.5% and 2 passes of 11% with intermediate heating up to 350° after every two reductions. With a decrease in thickness, billets cool faster. Therefore, 2 passes were carried out from one heating in order to withstand the deformation temperature range of 350°C–200°C. The billet thickness at the second stage varied in the range of 9.0–2.5 mm.

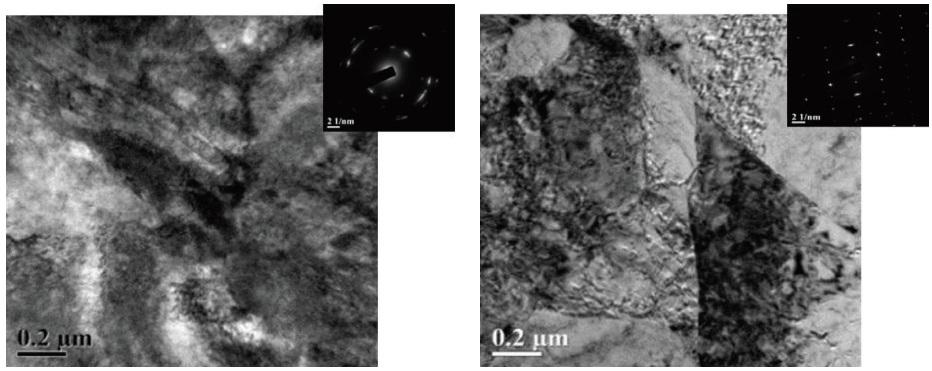
The total accumulated logarithmic degree of deformation at the second stage of rolling was $e2 = 1.28$, and from the beginning of rolling, $e12 = 2.19$. The change in the deformation degree in passage groups is shown in Figure 9.

At each stage of deformation, a part was cut off from the billet to analyse the properties after the deformation degree increased. The analysis of the microsections obtained from the billets showed that, at a rolling start temperature of 350°C, changes in the microstructure associated with recrystallization processes are imperceptible.

A further increase in the deformation degree at the second stage of deformation in the central part of the billet as a result of processing by TE and rolling formed a fine-grained structure with clear high-angle boundaries and cell sizes less than 100–200 nm (Figure 10(a)). In samples from the edge part of the rolled billet at the second stage of deformation, an inhomogeneous structure with low-angle deformation cells is formed (Figure 10(b)). The cell size is within 1 μm.

At the last, third stage (the billet thickness is from 2.5 to 1.2 mm), cross rolling of billets 50 mm long (Figure 11) was carried out in two different directions (see Figures 2, 3(a), and 3(b)). A change in the direction of rolling should activate slip bands with a new orientation and facilitate crystallite fragmentation [15]. From one heating, one reduction was carried out, while the billets (Figure 11(a)) were heated and brought to the mill in a muffle in order to reduce heat losses.

In the first case (Figure 11(b)), billets were rolled with heating up to 340°C. The deformation degrees along the passes were 8%, 13%, and 19%. When rolling relatively thin billets, the force and moment of rolling increase significantly; therefore, in the second case (Figure 11(c)), the billets were deformed at an initial heating temperature of 385°C. The furnace temperature during heating was 450°C. Deformation degrees in passes were 40% and 20%. As a result, billets with a thickness of 1.6 and 1.2 mm were obtained according to the first and second deformation options, respectively. The total deformation degree at the third stage during cross-rolling was 35% ($e = 0.43$) and 52% ($e = 0.73$) for the first and second cases.



(a) (b)

Figure 7: The microstructure of the billet after TE and the first stage of rolling ($h = 9.7$ mm, heating is 350°C): (a) rolling the central part of the sample after the TE; (b) rolling the edge part of the sample (without complete processing with TE).

Figure 8: The shape of the samples after the first stage of deformation and surface treatment. Workpiece dimensions: thickness is 9 mm, width is 10 mm, and length is 175 mm.

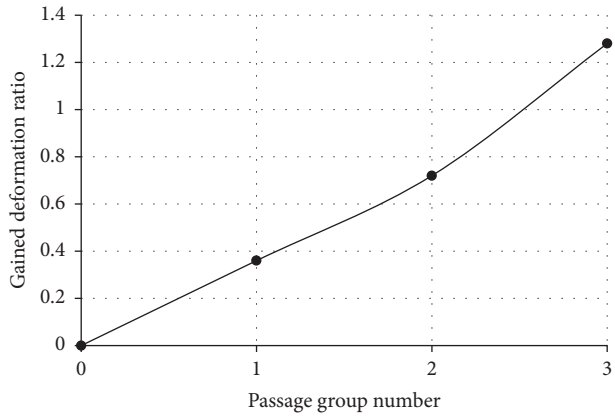


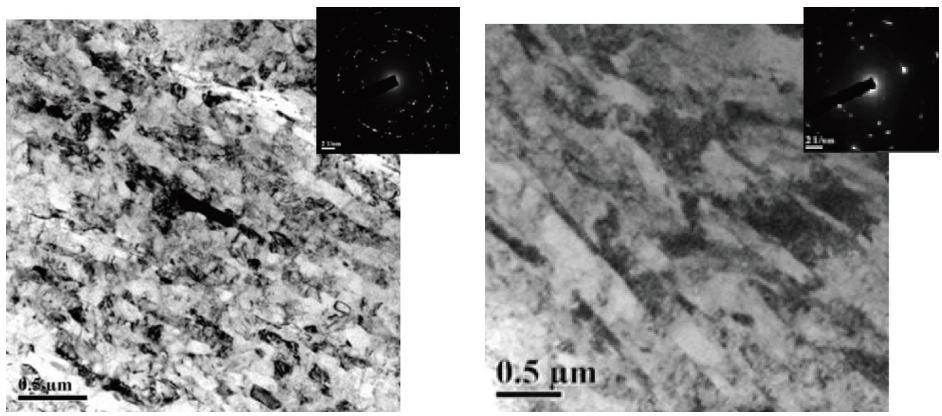
Figure 9: The change in the accumulated logarithmic degree of deformation at the second stage of billet rolling after TE from the passage group: rolling temperature range is 350°C–200°C; there are 2 reductions from one heating in the passage group; change in the billet thickness during rolling is 9.0–2.5 mm.

The microstructure of the rolled billet after the third stage of deformation at a temperature of 340°C (Figure 12) has no signs of the influence of the rolling thermal effects on its change. The analysis of the microstructure showed that after the third stage of deformation, a cellular structure with clear high-angle boundaries was formed in the material, and the size of its structural elements was 50–100 nm.

A similar deformation degree, but at a higher temperature of 385°C and with a simultaneous increase in the deformation degree per pass up to 40%, leads to an increase in the cell size up to 200 nm (Figure 13). The shape of the cells, which is close to the equiaxed one (Figures 13(a) and 13(b)), indicates the beginning of the dynamic recrystallization process. Therefore, the rolling temperature and the deformation degree in one pass must be regulated in order to maintain the billet structure obtained as a result of UFG processing.

Crystallite boundaries were studied to determine the effect of deformation on the boundaries' shape (Figure 14(a)). For a better visual presentation of information, the view of the boundaries of the selected area after the fast Fourier transform is shown in Figure 14(b).

To study the mechanical characteristics of titanium grade 1 with a UFG structure, flat samples were cut out from the billets rolled at different stages for tensile testing. The nature of the destruction of the samples

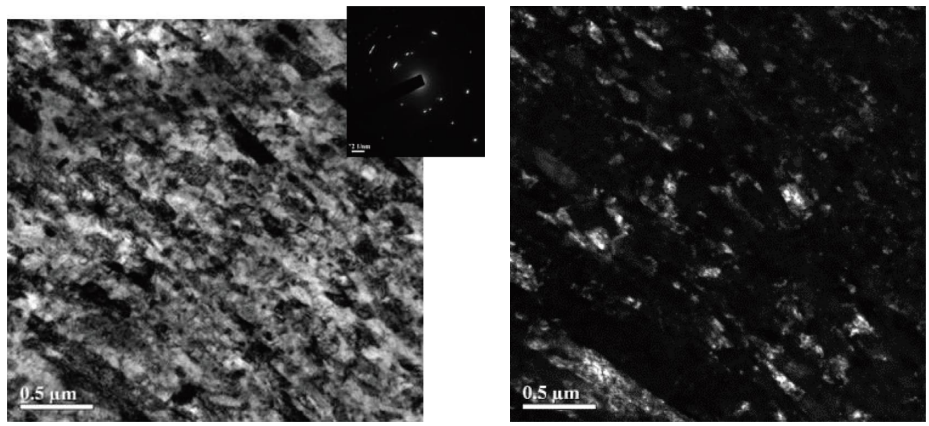


(a) (b)

Figure 10: The workpiece microstructure after the TE and the second stage of rolling ($h = 2.5$ mm, heating 350°C): (a) rolling after the TE; (b) rolling of the extreme part of the sample without complete processing by the TE.

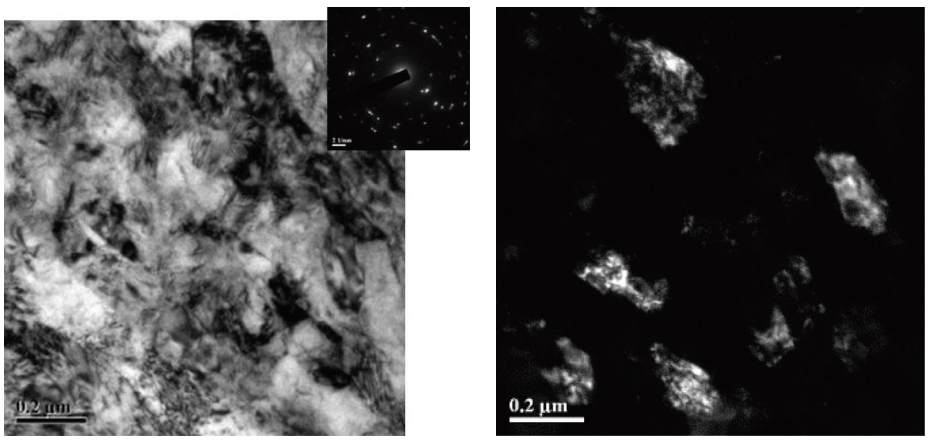
(a) (b) (c)

Figure 11: The shape of the samples: (a) after the second stage of deformation (dimensions: $2.5 \times 17 \times 50$ mm); after the third stage according to the TE and rolling mode: (b) at an initial temperature of 340°C, rolling in 3 passes (8%, 13%, and 19%), $h = 1.6$ mm; (c) at an initial temperature of 385°C, rolling in 2 passes (40% and 20%), $h = 1.2$ mm.



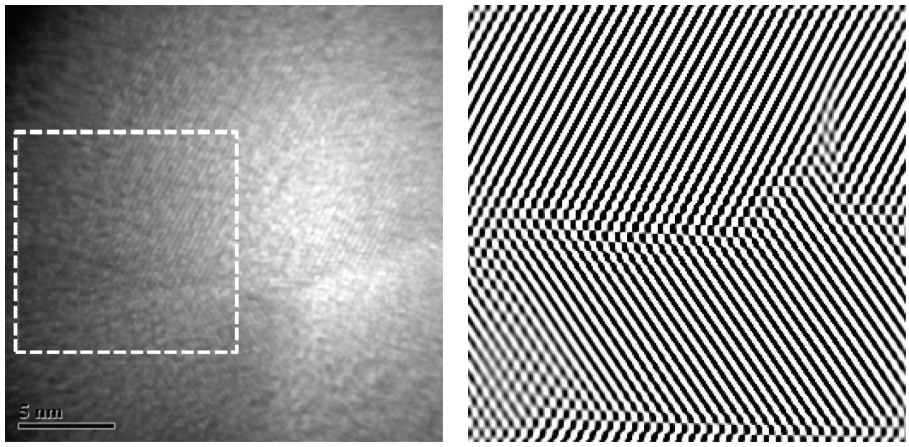
(a) (b)

Figure 12: Microstructure of the rolled billet after the third stage of deformation (heating 340°C, cross rolling of the sample, $h = 1.6$ mm): (a) bright-field image and microdiffraction; (b) darkfield image.



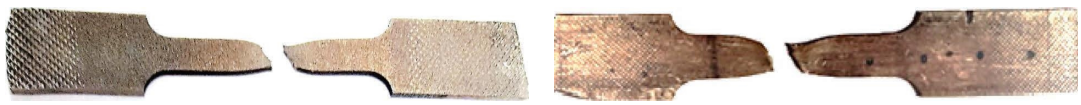
(a) (b)

Figure 13: Microstructure of the rolled billet after the third stage of deformation (heating 385°C, cross rolling of the sample, $h = 1.2$ mm): (a) bright-field image and microdiffraction; (b) darkfield image.



(a) (b)

Figure 14: Microstructure in the area of crystallite boundaries in the sample after the TE and the second stage of rolling ($h = 2.5$ mm, heating 350°C): (a) crystallite boundary fragment in the sample; (b) FFT processing of the selected area.



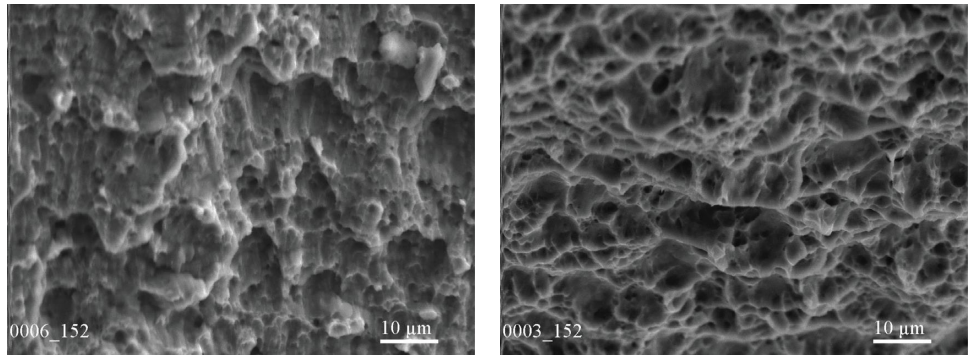
(a) (b)

Figure 15: The shape of the samples and the destruction nature: (a) after the first stage of processing ($h = 1.6$ mm); (b) after the second stage of processing ($h = 2.5$ mm).

under tension is shown in Figure 15. After the first stage of processing, the incoming billet with a thickness of 9 mm was cut into 4 samples with a thickness of 1.6 mm.

The fracture surface of rolled samples after tensile testing at a temperature of 20°C is shown in Figure 16. The crystallite size decreases with the deformation increasing. Crystalline decrease leads to a significant increase in mechanical characteristics. Strengthening also depends on the structure of crystalline boundaries. Dimple failure without delamination indicates a high strength of the crystalline boundaries.

The tensile curves of the samples obtained as a result of rolling are shown in Figure 17. The curves demonstrate the well-known fact that an increase in the deformation degree leads to an increase in the strength properties of



(a) (b)

Figure 16: Fracture surface of rolled samples after tensile testing at 20°C: (a) after the first stage of processing ($h = 1.6$ mm); (b) after the second stage of processing ($h = 2.5$ mm).

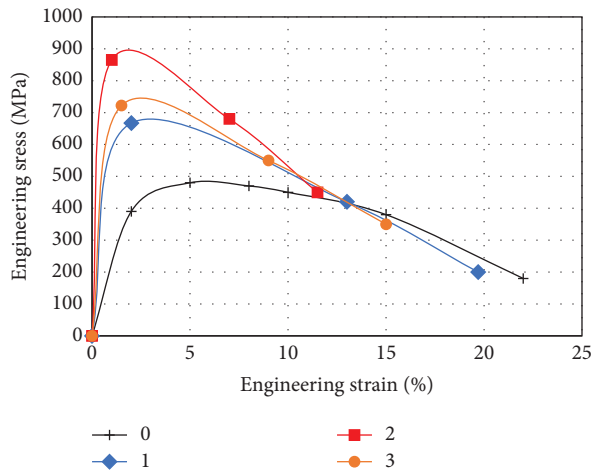


Figure 17: Tensile results of samples obtained after SPD processing by the TE method ($e = 4.6$) and rolling in the temperature range of 350–200°C: 0 indicates after the TE; 1 indicates after the 1st stage of rolling ($e = 0.91$); 2 indicates after the 2nd stage of rolling ($e = 2.19$); 3 indicates after the 3rd stage of cross rolling at 385°C ($e = 3$).

the billets with a relatively small decrease in ductility. The samples rolled at the third stage of deformation at a rolling start temperature of 385°C showed a decrease in strength compared to those processed at the second stage. This is caused by a change in the rolling conditions, namely, an increase in the processing temperature and the reduction degree, which affected the billet microstructure.

4. Conclusion

Experiments have shown that with the combined impact on the material, processes of constant change in the microstructure and billet properties occur. In the first stage, when using SPD by TE, the crystallite size decreases to <1 μm. To increase the billet length significantly, which is impossible on the basis of TE, subsequent rolling was carried out. The main goal during rolling was to at least preserve the resulting metal structure.

The condition for further refinement of the grains of the deformable material is to limit the deformation temperature and the deformation degree per pass, i.e., ensuring the crystallite's deformation by multiple rolling. In this experiment, the deformation degree in one pass was taken within 5 to 20%. The use of such deformation degrees made it possible to reduce the grain size in titanium grade 1 significantly. Subsequent rolling made it possible to reduce the size of structural elements to 50–100 nm and provide a significant increase in the mechanical characteristics of the billet material (up to 869 MPa) while maintaining satisfactory plasticity (up to 11.6%). At the same time, the selected temperature range of deformation should not lead to recrystallization processes in the material.

Increasing the reduction per pass reduces labor intensity and the number of heatings and increases the machining efficiency in whole. In doing so, it is necessary to set the limit processing modes. It was found that for titanium grade 1, an increase in the deformation degree in one pass during rolling up to 40% (to disrupt the structure transformation) and a simultaneous increase in temperature to 385°C led to a decrease in the quality of the UFG structure of the deformed material, which indicates the beginning of the process of dynamic recrystallization.

Data Availability

Data sharing is not applicable to this article as no datasets were generated or analysed during the current study.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors' Contributions

All authors participated in the design of this work and performed equally. All authors read and approved the final manuscript.

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