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EFFECT OF TEMPERATURE ON THE EVOLUTION OF STRUCTURE, CRYSTALLOGRAPHIC TEXTURE AND THE ANISOTROPY OF STRENGTH PROPERTIES IN THE Ti GRADE 4 ALLOY DURING CONTINUOUS ECAP

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Abstract. This paper presents the results of experimental studies on the evolution of structure, crystallographic texture and the anisotropy of strength properties in the volume of Ti Grade 4 billets subjected to continuous equal-channel angular pressing (ECAP). Continuous ECAP is performed at temperatures of 200 °C, 400 °C, and 450 °C, with 1 to 8 passes, using route B_C. The methods of X-ray diffraction analysis are employed to investigate the effect of processing regimes and strain degree during continuous ECAP on the structure of the material under study. As a result of computer modelling of the crystallograpic texture evolution, the regularities in the formation of preferred orientations and the anisotropy of strength properties are established, and the activity of various slip systems and twinning systems in titanium billets, depending on the number of ECAP passes, is evaluted. Estimation of the anisotropy of strength properties, based on building of yield contours, shows that an increase in the number of passes of continuous ECAP promotes the formation of a more isotropic structure. The obtained results allow to explain and predict the deformation behavior of nanostructured Ti Grade 4 with consideration of its microstructure and crystallographic texture parameters.

1. INTRODUCTION

To date, the techniques of severe plastic deformation (SPD) have been actively developed, such as equal-channel angular pressing (ECAP) and high pressure torsion (HPT) [1-3]. These techniques enable formation of bulk nanostructured and ultrafine-grained (UFG) states in billets of various metals and alloys. These states are characterized by attractive properties and a high potential for industrial application. Of special interest are the extraordinary mechanical properties of bulk nanostructured materials. It has been shown [1] that materials subjected to SPD may exhibit very high strength combined with sufficient ductility, as well as high fatigue strength, low-temperature or high-strain-rate superplasticity. A characteristic feature of bulk nanostructured materials produced by SPD is an extremely small grain size, the presence of a high density of crystalline structure defects at grain boundaries and their very non-equlibrium state [1-3].

With a view to produce bulk nanostructured billets for industrial application and increase the efficiency of these techniques, several new procedures have been proposed by the researchers at Ufa State Aviation Technical University (USATU). Among these procedures are ECAP with parallel channels and continuous ECAP [3]. Of special interest, from the practical standpoint, is continuous ECAP, since this

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procedure allows ro produce long-length rods with improved mechanical properties [3].

It is known that temperature is one of the most important parameters of plastic straining, also during SPD processing [1,2]. A decrease in temperature may activate new slip systems, twinning systems, block the mechanisms of dislocation climb. The processes of dynamic recovery and recrystallization become impossible at low temperatures.

Continuous ECAP is a rather new process, and in this connection the effect of the processing temperature on the character of structural components refinement and on the operating slip systems, as well as twinning systems, still remains unstudied. The conventional ECAP of Ti Grade 4 is usually conducted at rather high temperatures (400 °C) [1,2]. In this respect, of great current interest is a study on the possibility of decreasing the ECAP processing temperature, including the temperature of continuous ECAP, which would allow to increase the efficiency of this technique. At the same time, one should bear in mind that at temperatures that are too low, problems emerge associated with the need to apply very high straining forces, with the durability of the die-set and the ductility of the material under processing.

X-ray diffraction analysis (XRD), which provides an opportunity to estimate the size of coherent scattering domains (CSD), the value of the crystal lattice elastic microdistortions, the dislocation density, the activity of the operating slip and twinning systems, and so on, is the essential tool for investigation of bulk nanostructured materials. Using XRD, it is possible to successfully control changes in the microstructure and establish the mechanisms leading to such chages [4].

Crystallographic slip and deformation twinning determine the character of the preferred grain orientation, i.e. crystallographic texture, in metallic materials [5]. Analysis of the forming crystallographic textures, using modelling, is an important tool for investigation of the mechanisms of plastic deformation in metals and alloys [5].

The results of experimental studies on Ti processed for the first pass of conventional ECAP at 450 °C showed that there was a large number of twins in the structure [6]. This allowed to conclude that during the first pass of ECAP Ti is deformed primarily by twinning along $\{10\bar{1}1\}$ planes. Paper [7] presented the results of microstructural studies of Ti samples after the first pass of ECAP conducted at temperatures of 200 °C to 600 °C. It was established there that the pressing temperature had a noticeable effect on the operating deformation

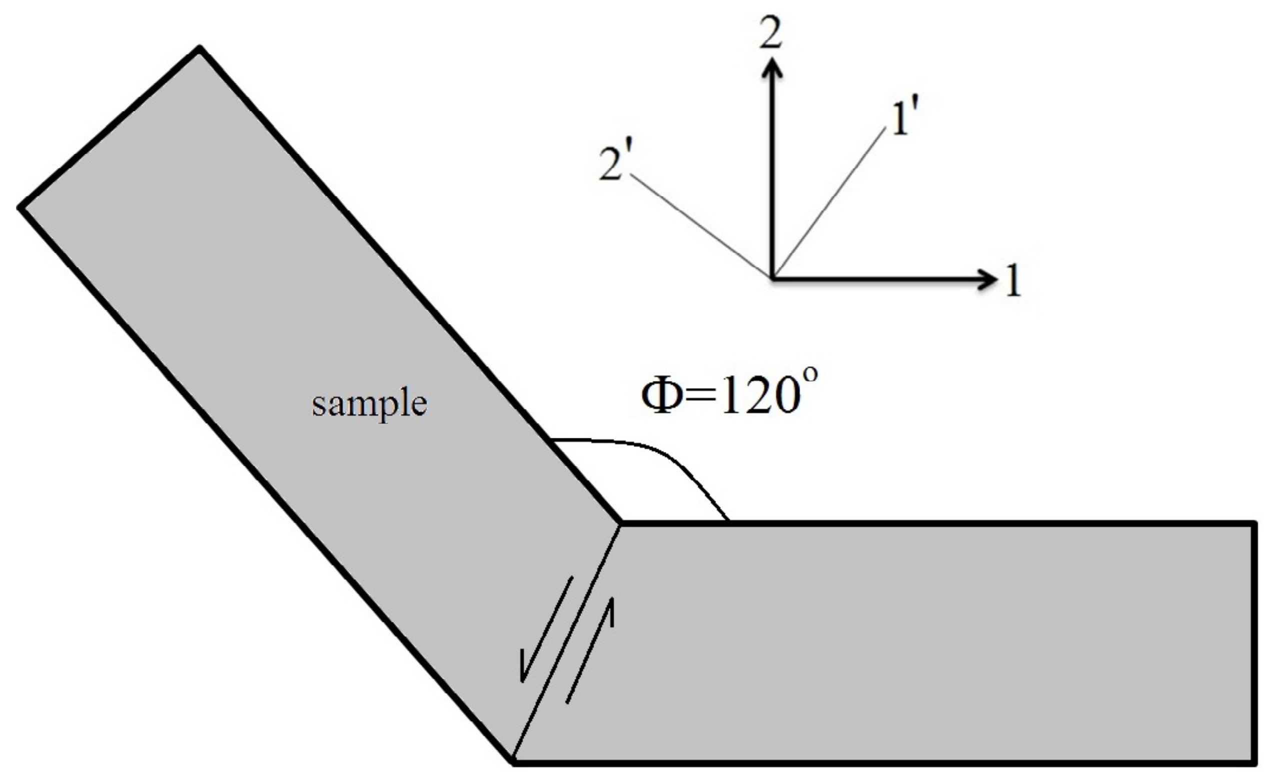

Fig. 1. Schematic illustration of the continuous ECAP process and the used coordinate system.

mechanisms. In particular, the TEM studies revealed that deformation by twinning becomes visibly active in the temperature range from 200 °C to 400 °C, but a further temperature increase up to 600 °C leads to a marked decline in the role of twinning and the activation of dislocation glide [7].

The aim of the present work is to study the effect of temperature and the number of passes during continuous ECAP on the character of microstructure evolution and on the mechanisms providing for material flow in the course of continuous ECAP, as well as to identify the structural parameters responsible for formation of the high-strength state and its anisotropy.

2. MATERIALS AND METHODS OF THE EXPERIMENT AND MODELLING

Ti Grade 4 with an average grain size of 10 μm was selected as the initial material. The initial billets of as-annealed Ti had the shape of a rectanular parallelepiped with a length of 25 cm and a square base with an edge of 11 mm. The billets were subjected to continuous ECAP at temperatures of 200 °C, 400 °C, and 450 °C. Different degrees of accumulated strain were attained by a repetitive pressing of the billets through two channels intersecting at an angle of Φ=120° (Fig. 1).

Continuous ECAP was conducted using route B_C where the sample was rotated between passes by 90° in respect to the billet longitidinal axis (axis 1).

X-ray diffraction analysis was conducted employing a Rigaku Ultima IV X-ray diffractometer. During this process, Cu Kα1 radiation (λ=0.154060 nm) was used at a voltage of 45 kV and a current of 30 mA. High-resolution images of the standing out X-ray peaks were taken with a step of 0.01° and a counting time of 10 s per step. A quantitative assessment was made of the CSD size *D*, of the value of the crystal lattice elastic microdistortions $<\varepsilon^2>^{1/2}$, using the PDXL software suite (www.rigaku.com), and the dislocation density ρ was estimated according to the method described in [8]. The texture formation processes in Ti Grade 4 were analyzed using a DRON-3m X-ray diffractometer equipped with an automated texture attachment. When taking images of pole figures (PFs), filtered Cu $K_{\alpha1}$ radiation (0.1540598 nm) was used. The reflections were filmed within the variation in the radial angle γ from 0° to 75° and the azimuth angle δ from 0° to 360°.

The diameter of the area subjected to radiation was 0.6 mm. The investigation was performed in the geometrical center of the billet's cross section (plane 2-1) (Fig. 1). As a result, a set of intensities of the reflected X-rays was obtained. The orientation distribution function (ODF) and complete PFs were built on the basis of the results of filming of incomplete pole figs. for the planes $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$. The results of the experimental studies were presented in the form of complete PFs calculated using the software suite LaboTEX (www.labosoft.com.pl).

Modelling of the texture formation processes was conducted in the framework of the viscous-plastic self-consistent (VPSC) model [9]. To reveal the active slip systems responsible for appearance of particular maxima on the PFs, seven slip and twinning systems, most typical for Ti with an HCP lattice, were selected on the basis of literature analysis. These systems include: basal $\{0001\}<\bar{1}\bar{1}20>$, prismatic $\{10\bar{1}0\}<\bar{1}2\bar{1}0>$, first-order pyramidal $\{10\bar{1}1\}<\bar{1}2\bar{1}0>$, first-order pyramidal $\{10\bar{1}1\}<\bar{1}\bar{1}23>$ and second-order pyramidal $\{11\bar{2}2\}<\bar{1}\bar{1}23>$ slip systems, as well as tensile $\{10\bar{1}2\}<\bar{1}011>$ compression $\{2\bar{1}\bar{1}2\}<2\bar{1}\bar{1}\bar{3}>$ twinning systems.

3. RESULTS AND DISCUSSION

A comparative XRD analysis indicates that the CSD sizes, the crystal lattice elastic microdistortions, as well as the dislocation density for nanostructured states produced by continuous ECAP at 200 °C, 400 °C and 450 °C, differ significantly from those for the coarse-crystalline state (Table 1).

After processing by continuous ECAP for 8 passes at T=450 °C, the CSD size decreases approximately twofold, as compared with the coarse-crystalline state, and equals 32±3 nm, which is not contradictory to the results obtained in [8] for CP Ti (grade 2). At 450 °C the reduction in the SCD size is smaller than that at 200 °C and 400 °C, and its dependence on the number of passes varies with regularity (Table 1). The value of the root-mean-square microdistortions of the crystal lattice after a large number of passes reaches saturation, and so does the dislocation density.

As the temperature of continuous ECAP is decreased to 400 °C, there is a consistent reduction in the CSD size with increasing number of passes. Here the levels of root-mean-square microdistortions monotonously grow, and the dislocation density grows as well. The level of the root-mean-square microdistortions of the crystal lattice after the first pass of continuous ECAP at 400 °C is about two times higher, and after 8 passes – about 4.2 times higher than the corresponding value for the coarse-crystalline state. At the same time, as a result of processing by continuous ECAP for 8 passes at 400 °C, the dislocation density grows more than tenfold, which indicates a high density of intoduced defects. However, this value of dislocation density (Table 1) is slightly higher than that for pure Ti after 8 passes of ECAP at a temperature of 450 °C with subsequent cold rolling ($\rho \sim 3 \cdot 10^{15}$ m^{-2}) [10]. This difference is apparently associated with the purity of the compared materials, strain degree and temperature, different deformation schemes.

As the temperature of continuous ECAP is decreased to 200 °C, there is also a consistent reduction in the CSD size with increasing number of passes. Here the levels of root-mean-square microdistortions monotonously grow, and the dislo-

Table 1. Structure parameters of titanium billets (grade 4) produced by continuous ECAP.

State	CSD, nm			$<\varepsilon^2>^{1/2}$, $\times 10^{-4}$			ρ, 10^{15} m^{-2}		
	200 ℃	400 ℃	450 ℃	200 °C	400 °C	450 °C	200 ℃	400 ℃	450 ℃
Initial	69.5±11			7.81±1.02			~0.23		
1st pass	32±3	38±4	43±4	19.11 ±1.93	17.15 ±1.49	16.47±1.36	~0.89	~0.73	~0.65
2nd pass	29±3	34±3	41±4	21.54±2.09	18.88 ±1.79	17.69±1.64	~1.87	~1.65	~1.51
4th pass	26±3	32±3	39±4	28.23 ±2.16	26.97±2.14	23.53±2.12	~3.27	~2.99	~2.71
6th pass	25±2	33±2	35±3	33.17 ±2.31	29.27±2.36	26.71±2.16	~5.33	~4.94	~4.79
8th pass	25±2	28±2	32±3	34.56 ±2.43	32.81±2.40	29.89±2.41	~5.72	~5.27	~5.09

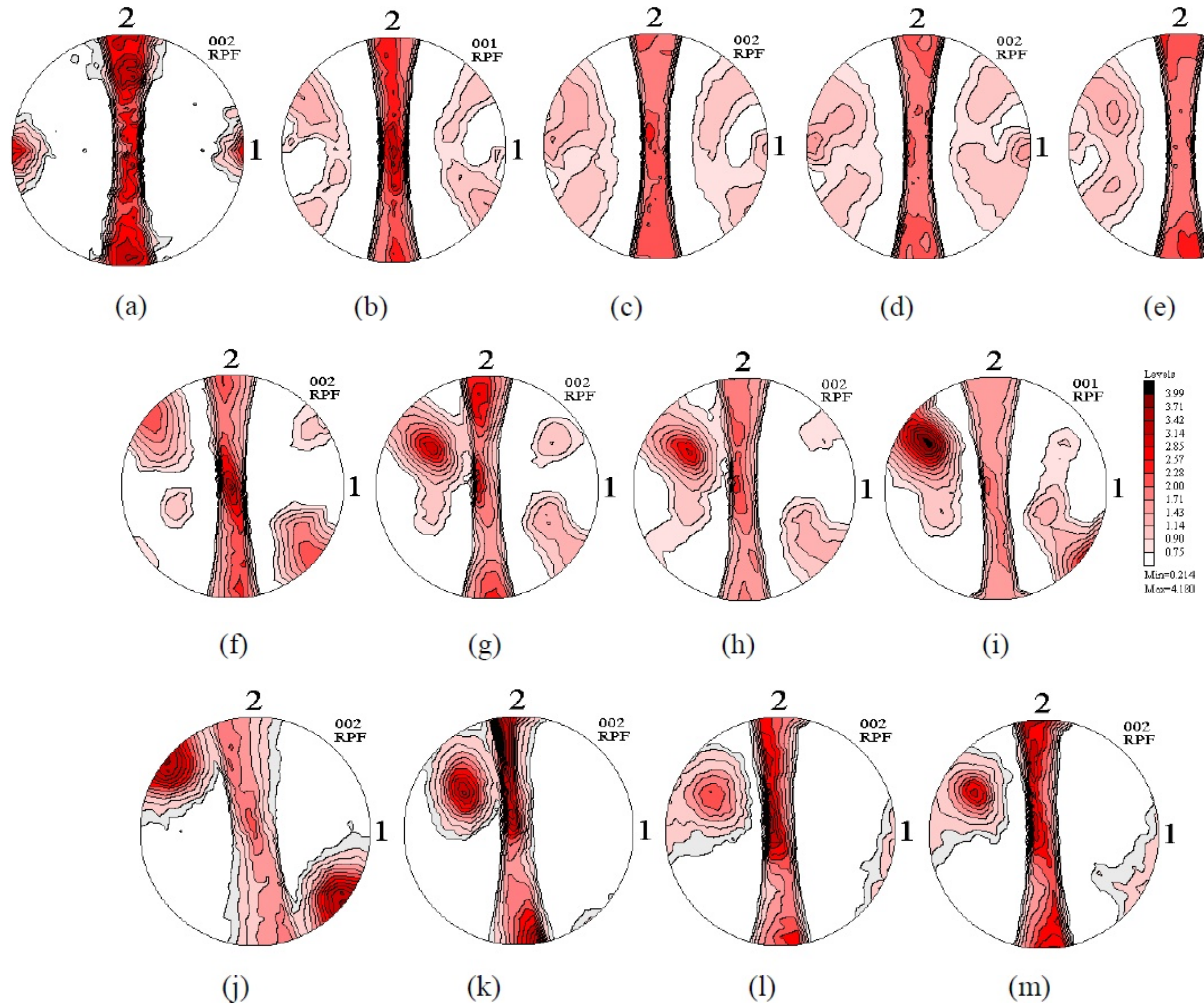

Fig. 2. Experimental PFs (0002) for Ti Grade 4 in various states. Longitudinal section. Initial state (a), 1, 2, 4 and 8 passes 450 °C (b)-(e), 1, 2, 4, and 8 pases 400 °C (f)-(i), 1, 2, 4 and 8 passes 200 °C (j)-(m).

cation density grows as well. The level of the root-mean-square microdistortions of the crystal lattice after the first pass of continuous ECAP at 200 °C is 2.44 higher, and after 8 passes – about 4.42 times higher than the corresponding value for the coarse-crystalline state. At the same time, as a result of processing by continuous ECAP for 8 passes at 200 °C, the dislocation density grows more than six times, which indcates a high density of intoduced defects.

Thus, the X-ray diffraction analysis shows that a decrease in the temperature of continuous ECAP contributes to additional microstructure refinement.

The experimental complete PFs (0002) for Ti (grade 4) in various investigated states (the intial state, the states after the first, second, fourth and eighth passes of continuous ECAP at a temperature of 450 °C) are displayed in Fig. 2a. At the periphery of the PF in the initial state two texture maxima are observed, related to orientations of the type {0001}<$\bar{1}\bar{1}20$>. In the center of the PF there is a fiber {01$\bar{1}$0}<*uvtw*> oriented along the PF vertical axis (Fig. 2a). The location of texture maxima in the PF (0001) in the initial state (Fig. 2a) is similar to the one observed during rolling [5] and is conditioned by the thermomechanical process of the initial rod fabrication.

The experimental PF (0002) after the first pass of continuous ECAP at *T* = 450 °C (Fig. 2b) is characterized by four main maxima located at the PF periphery at angles approximately equal to 60° in respect to axis 1 which corresponds to the orientation of the billet longitudinal axis. The location of the texture maxima is similar to the one observed during conventional ECAP (*T* = 400 °C) with the angles of channels intersection equal to 120°. Such a texture can be characterized using the ideal orientations $H1_\theta$, $H2_\theta$, $H3_\theta$, $H4_\theta$, $H5_\theta$, and $H6_\theta$, corresponding to the texture of simple shear (Fig. 3) [11]. The maxima indicate the activity of pyramidal (orientations $H2_\theta$, $H3_\theta$, $H4_\theta$) and basal (orientation $H1_\theta$) slip systems.

Besides, in the center of the PF another maximum is observed, corresponding to the orientation $H2_\theta$ (0001)<$\bar{1}010$> (Figs. 2b and 3). The character of location of all the texture maxima in the PF (0002) indicates that the basal planes are mostly set parallel to the shear plane (the plane of the channels intersection). Also, they are favorably oriented for the dislocation glide <*a*> along this plane, as well

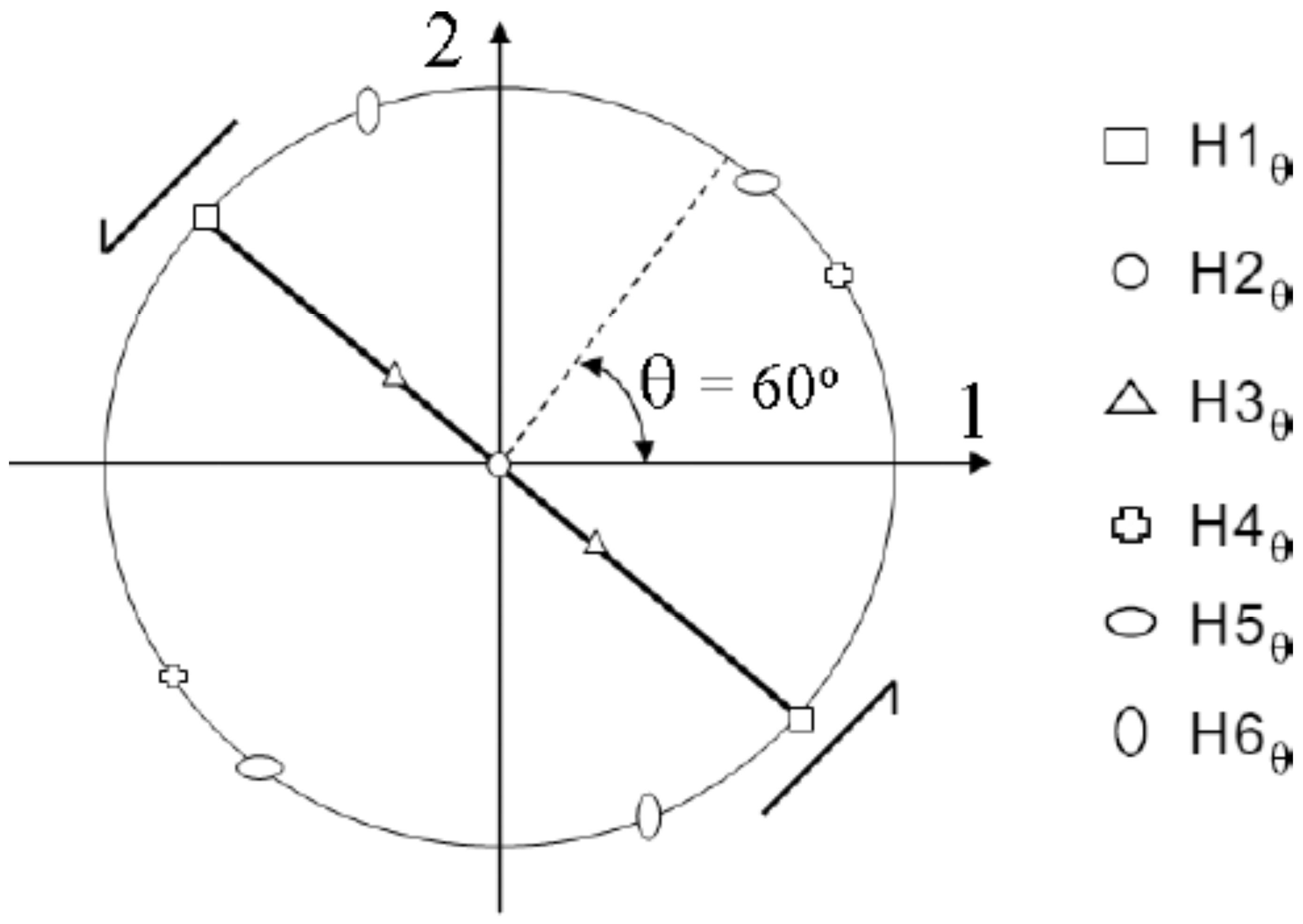

Fig. 3. Locations of the ideal orientations of simple shear during ECAP in the PF (0002) for metals with an HCP-lattice (c/a = 1.633) [11]. The angle of channels intersection is 120°.

as along the first-order and second-order pyramidal planes.

After the second, fourth and eighth passes the appearance of the PF practically does not change, and it looks similar to the PF after the first pass (Fig. 2b). At the same time, after the eighth pass (Fig. 2e) the orientation ($\bar{1}2\bar{1}1$)<31$\bar{4}$1>, which is close to the PF center and belongs to the pyramidal type, becomes stronger.

The experimental PF (0002), after the fist pass of continuous ECAP at T = 400 °C (Fig. 2f), is characterized by two main maxima located at the PF periphery, and is, on the whole, similar to the one at T = 450 °C. The location of the texture maxima can be described using the ideal orientations $H1_\theta$, $H2_\theta$, $H3_\theta$, $H4_\theta$, $H5_\theta$, and $H6_\theta$, corresponding to the texture of simple shear (Fig. 3). The maxima indicate the activity of basal and pyramidal slip systems in the shear pane (orientation $H1_\theta$), as well as twinning systems (orientation $H6_\theta$).

After the second pass of continuous ECAP at T = 400 °C, the crystallographic orientations of the grains are different from the ones typical for the state after the first pass. The evidence of this is the change in the texture maxima location (Fig. 2g). The basal plane which was parallel to the shear plane (perpendicular to plane 1-2) after the first pass, is rotated by ~30 degrees in relation to axis 2. This leads to the appearance of a new orientation (01$\bar{1}\bar{1}$) <$\bar{2}$111> displaced to the PF center.

After the fourth and eighth passes of continuous ECAP at T = 400 °C, the appearance of the PF practically does not change, and it looks similar to the PF after the second pass (Fig. 2g). At the same time, after the fourth pass the orientation $H2_\theta$ (0001)<$\bar{1}$010> in the PF center becomes weaker (Fig. 2d). Besides, the pole density of the orientation ($\bar{1}2\bar{1}1$)<31$\bar{4}$1>, which is close to the PF center and belongs to the pyramidal type, has a tendency for insignificant growth with increasing number of ECAP passes.

The experimental complete PFs (0002) of Ti Grade 4 after the first, second, fourth and eighth passes of continuous ECAP at a temperature of 200 °C are displayed in Fig. 2j-2m. On the whole, the PFs in different structural states at a temperature of 200 °C are similar to the ones obtained at a temperature of 400 °C. However, the pole density of the orientation (01$\bar{1}\bar{1}$)<$\bar{2}$111>, which belongs to the pyramidal type, decreases with increasing number of continuous ECAP-C passes, while the «twin orientation» $H6_\theta$ (12$\bar{3}$0) <0001> disappears (Figs. 2j-2m).

Interpretation of the obtained experimental crystallographic textures is rather complicated, since they do not allow to establish the operating slip and twinning systems. In this connection, an analysis of the texture formation processes in the course of SPD processing was conducted using computer modelling within the viscous-plastic consistent model. Fig. 4 displays the results of the modelling of the texture formation processes during the processing of Ti Grade 4 by continuous ECAP, when the selected slip and twinning systems are active. The direct PFs (0001), resulting from modelling, reproduce the main texture maxma $H1_\theta$, $H2_\theta$, $H3_\theta$, $H4_\theta$,

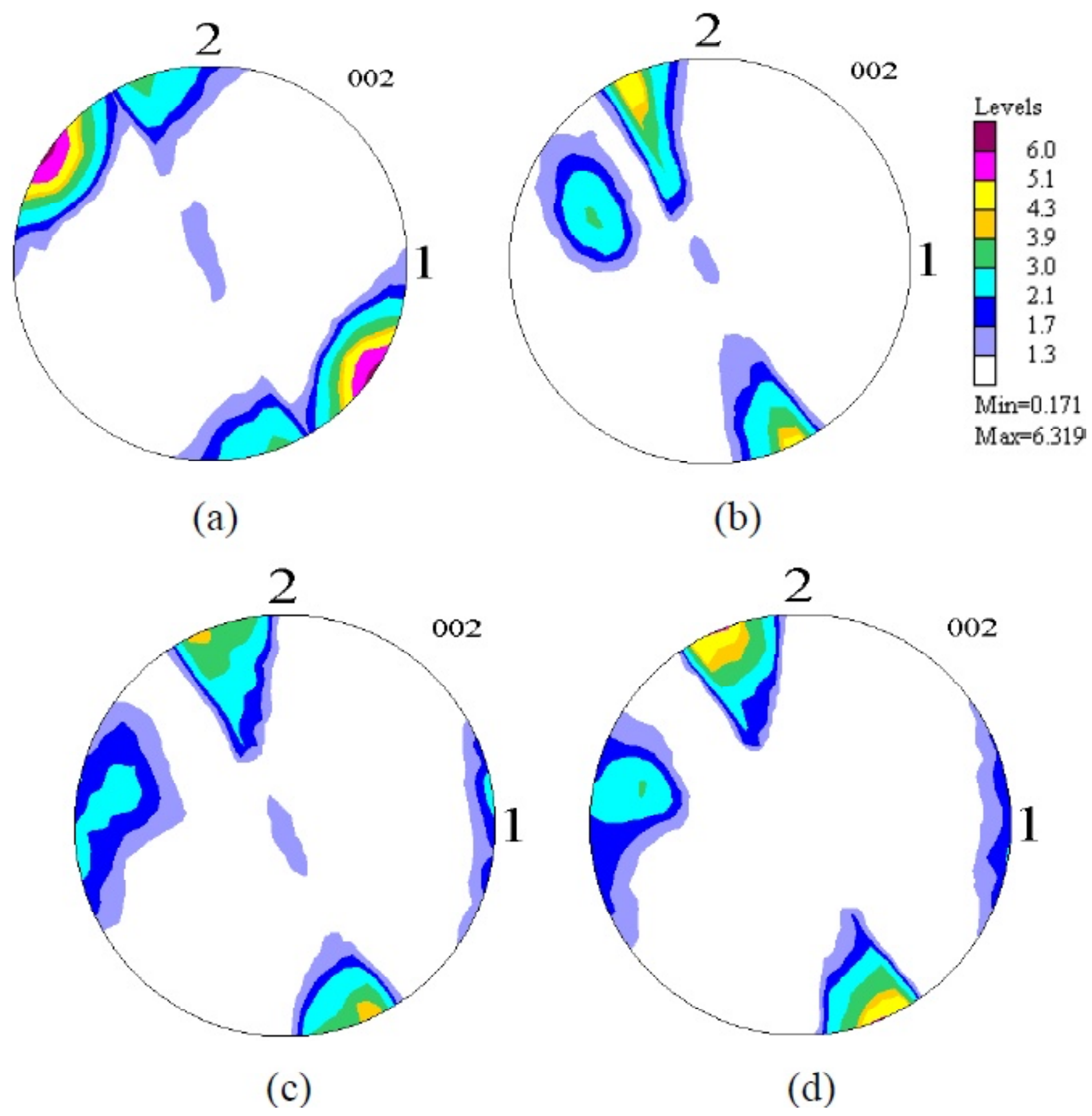

Fig. 4. Model PFs (0002) of Ti Grade 4 in plane 1-2 after processing by continuous ECAP through different numbers of passes: 1 pass (a), 2 passes (b), 4 passes (c), 8 passes (d). Longitudinal section. T = 200 °C.

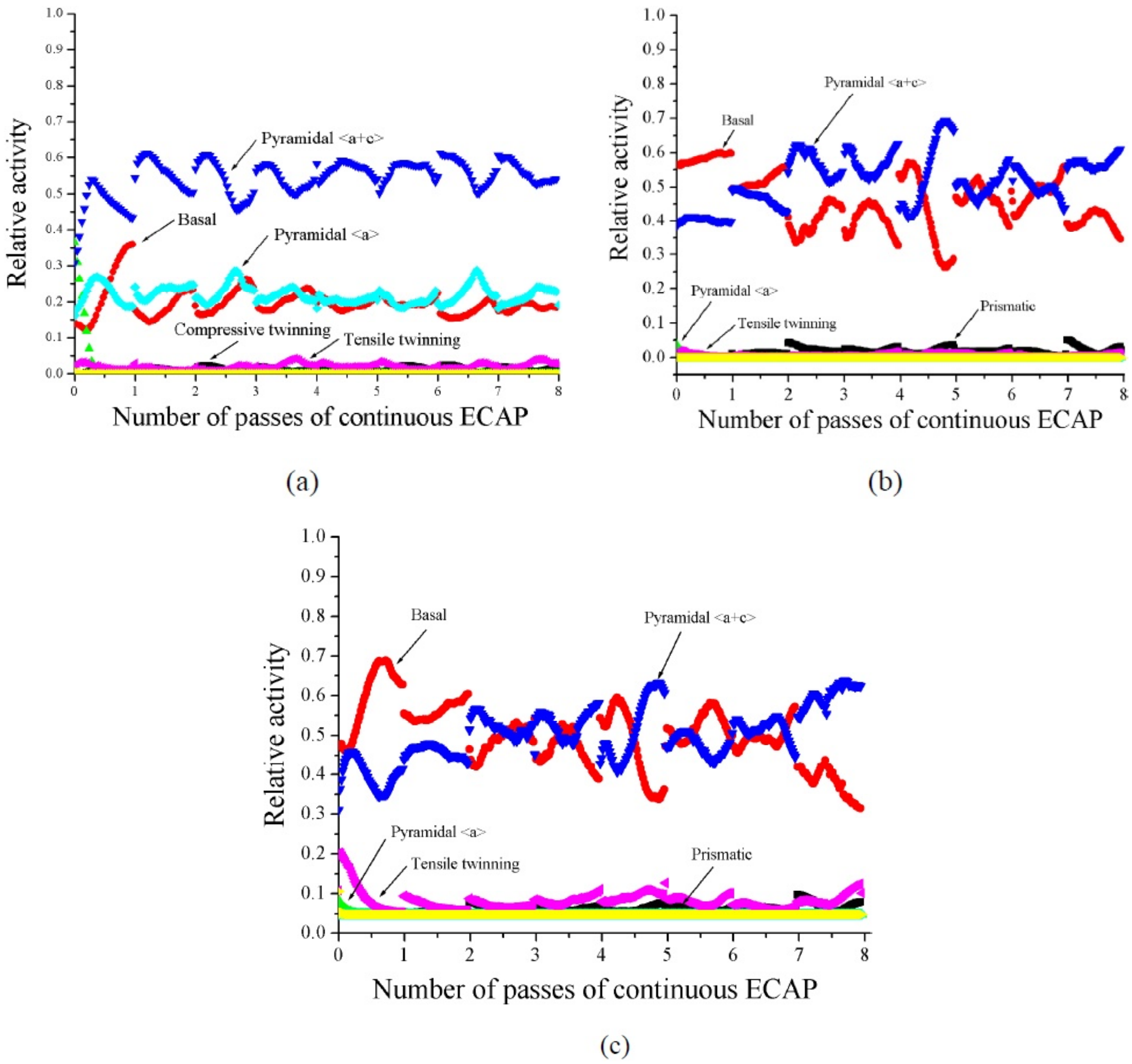

Fig. 5. Relative activity of the slip and twinning systems depending on the number of passes of continuous ECAP. Ti Grade 4. *T* = 450 °C (a), *T* = 400 °C (b), *T* = 200 °C (c).

$H5_\theta$, and $H6_\theta$ of the shear texture (Fig. 3). Comparison of the experimental results (Fig. 2) and modelling results (Fig. 4) in the corresponding states (*T* = 200 °C) demonstrates a good correlation between them.

Fig. 5 presents the results of the modelling of the texture formation processes at temperatures of 450 °C, 400 °C, and 200 °C with account of the slip and twinning systems selected on the basis of literature data and corresponding to the given temperature of the critical shear stress. During continuous ECAP conducted at *T* = 450 °C, the most active systems, as compared to the other slip and twinning systems, are pyramidal <*c+a*> $\{10\bar{1}1\}\langle\bar{1}\bar{1}23\rangle$ (first-order), basal {0001} and pyramidal <*a*> $\{10\bar{1}1\}\langle\bar{1}2\bar{1}0\rangle$ slip systems (Fig. 5a). The activity of dislocation glide via basal systems has a tendency to decrease with increasing accumulated strain during the first pass of continuous ECAP. The processes of tensile and compression twinning in the course of continuous ECAP are restricted (Fig. 5a).

During the first pass of continuous ECAP conducted at *T* = 400 °C, the most active systems, as compared to the other slip and twinning systems, are basal {0001} slip systems (Fig. 5b). The activity of dislocation glide via basal systems has a tendency to increase with increasing accumulated strain during the first pass of continuous ECAP. Besides, during the first pass, pyramidal <*c+a*> $\{10\bar{1}1\}\langle\bar{1}\bar{1}23\rangle$ (first-order) slip systems are also active, their action having a non-monotonous character.

The processes of tensile and compression twinning are also under way during the first pass of continuous ECAP at *T* = 400 °C, but their activity is rather low (Fig. 5b). Here, the processes of twinning via the systems $\{10\bar{1}2\}\langle\bar{1}011\rangle$, which are active at the beginning of the first pass, get impeded at the end of the first pass. As the number of the continuous ECAP passes increases, the activity of particular slip and twinning systems changes consistently (Fig. 5a). At the initial stages of the pressing, the basal and pyramidal <*a+c*> slip systems of

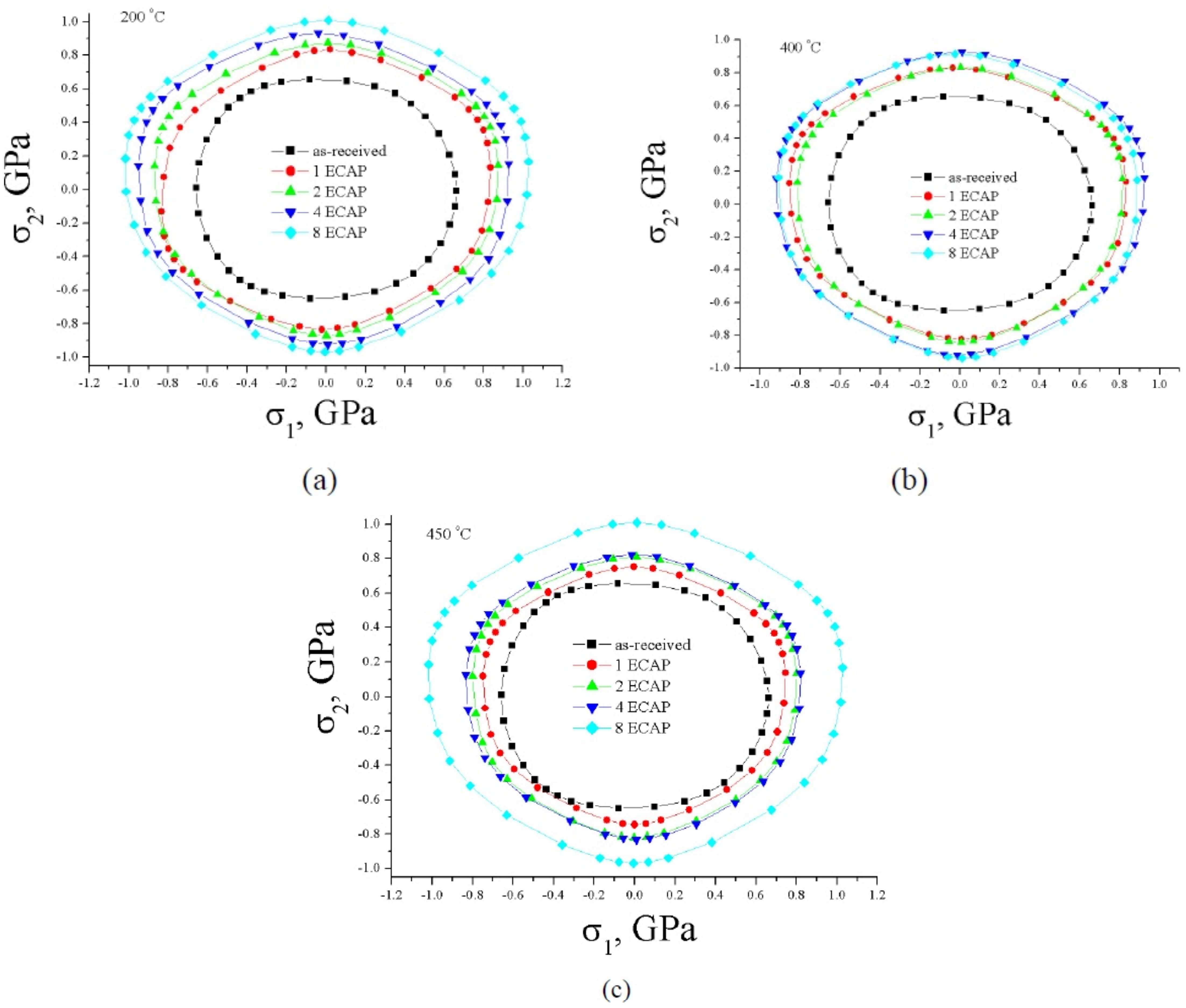

Fig. 6. Yield contours for Ti Grade 4 in various states: Longitudinal section. σ_1 – coincides with direction 1, σ_2 – coincides with direction 2. Continuous ECAP at *T* = 450 °C (a), *T* = 400 °C (b), *T* = 200 °C (c).

the first order are the most active. By the end of the respective pass the fraction of basal slip systems is gradually reduced and, in contrast, the fraction of pyramidal slip system grows. In addition, starting from the second pass, prismatic slip systems become slightly activated (Fig. 5a). A similar tendency was observed in the works [12,13] which demonstrated that the most active slip systems are basal, prismatic and pyramidal systems.

The results of the assessment of particular slip and twinning systems at *T* = 200 °C are presented in Fig. 5c. As a result of a decrease in the pressing temperature, the twinning processes intensify (Fig. 5c). It can be seen from Fig. 5c that at the beginning of the first pass of continuous ECAP at *T* = 200 °C twinning systems become activated, which in a natural manner leads to microstructure refinement. The processes of deformation by twinning are also active at large strains of severe plastic deformation (Fig. 5c). At the same time, the fraction of active dislocations moving via basal and pyramidal <*a*+*c*> slip systems of the first order becomes lower than that realized at *T* = 400 °C. Also, worth mentioning is an insignificant reduction in the fraction of prismatic systems with decreasing temperature of processing by continuous ECAP with large strains.

It was demonstrated earlier that the activity of basal, pyramidal and prismatic slip systems is dominant in the formation of crystallographic texture in Ti Grade 2 in the process of conventional ECAP [14]. At the same time, it was established that the role of twinning is noticeable only at the beginning of the first pass of ECAP in the considered conditions (*T* = 450 °C, the pressing speed is 6 mm/s). A comparative analysis showed that in the process of both conventional and continuous ECAP, the same slip and twinning systems become activated.

Fig. 6 displays the results from the modelling of yield stress anisotropy along various directions in the billet. The calculations were performed using the software suite LaPP (Los Alamos Polycrystal Plasticity) [5]. The dots in the graphs denote the yield stresses ($\sigma_{0.2}$) along various directions in the billet. Here axis σ_1 coincides with the longitudinal direction (axis 1 in Fig. 1) in the billet, while axis σ_2 coincides with the direction perpendicular to the longitudinal direction (axis 2 in Fig. 1). The modelling results indicate that, at all the temperatures under consideration, an increase in the number of continuous ECAP passes should contribute to the formation of a more isotropic state.

3. CONCLUSIONS

As a result of the conducted experimental studies, using X-ray diffraction analysis and computer modelling, the effect of the continuous ECAP temperature on the character of microstructure evolution, the operating deformation mechanisms, the level and anisotropy of strength properties in Ti Grade 4 has been investigated. It has been established that at a temperature of 450 °C occurs a consistent decrease in the size of grains and CSD. Also, with increasing number of passes, a monotonous growth of root-mean-square microdistortions and dislocation density is observed.

As the processing temperature is decreased to 200 °C, activation of the twinning processes, which contribute to microstructure refinement, is observed. Here the values of root-mean-square microdistortions and dislocation density in the crystal lattice reach an extremely high level.

As a result of the computer modelling of the crystallographic texture evolution, it has been demonstated that in the process of continuous ECAP, at all the temperatures under consideration, the most active systems, as compared to the other slip and twinning systems, are basal {0001} and pyramidal <*a*+*c*> slip systems of the first order. Also, the formed crystallographic texture can be characterized by the ideal orientations $H1_\theta$, $H2_\theta$, $H3_\theta$, $H4_\theta$, $H5_\theta$, and $H6_\theta$, corresponding to the texture of simple shear. An increase in the number of continuous ECAP passes leads to the restriction of twinning processes, as well as to intensification of prismatic slip systems.

The anisotropy of strength properties becomes less pronounced with increasing number of continuous ECAP passes and with increasing processing temperature.

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