EFFECTS OF CROSS ROLLING AND STRESS RELAXATION ON TEXTURE OF Zr-2.5%Nb PLATES

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An effect of cold rolling on texture and the Kearns' texture parameter (TP) of Zr-2.5%Nb alloy plates, made by straightening the rings of the ∅15.0×1.5 mm tube, has been investigated by the X-ray method of inverse pole figures (IPFs). The rolling was carried out in a direction perpendicular to the direction of longitudinal rolling of the original tube, and did with deformations from 7 to 55% with intervals of ~ 15...40 min between the passages. IPFs and a plot of TP changes with the deformation degrees on the plate's surfaces are built. Strong oscillations of the graph are noted. By investigation of the samples etched with 30 μm, as well as original samples, selectively processed without delays, it has been established that this is due to the surface stresses relaxation in the intervals between rolling acts. For the continuous rolling process and the conditions of uniformity of the initial texture of the alloy, the graph of TP changes has been determined. The staged nature of the TP dependence on deformation is revealed – existence of an initial stage of almost linear growth and the subsequent stage of insignificant changes.

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INTRODUCTION

This work is a part of the cycle of studies of patterns of texture changes during cold deformation of nuclear materials. These are metals with a crystalline hcp lattice, namely, hafnium, zirconium and its alloys. The work is related to tasks of analysis, control and predicting texture characteristics of products of reactor designation of these metals, optimization of their exploitation characteristics. The purpose of this series of works is to study regularities and mechanisms of texture changes in products of the listed metals.

A feature of the work is, first of all, in choice of the approach for textural research – this is use of the Kearns' texture parameter (TP, [1]). Mechanical, thermophysical and other tensor parameters are directly related to it in different geometric directions of products made of metals with crystallographic anisotropy [2]. Such characteristics are the coefficient of thermal expansion, thermal and electrical conductivity, orientation of precipitates, elastic modules etc. TP plays an important role in behavior of these materials under irradiation – this is resistance to radiation growth of constructions from these materials during reactor operation.

Use in principle of TP as the subject of analysis should bring single-valued relationships in the study of texture and structural patterns of deformation. In particular, based on texture studies of hafnium and Zr-2.5%Nb alloy, it was shown [3, 4] that, with a low initial texture, these metals can have an initial stage of significant changes in TP with the degree of rolling. The next stage is characterized by relative moderation. This staged character occurs most visible for the TP measured in the rolling direction of Hf and Zr plates [3, 4]. When studying plates with a high initial texture, the initial stage of TP would practically not be detected.

It follows from these results that both the TP changes and the length of the initial stage of longitudinal rolling of Zr and Zr-2.5% Nb alloy can be unambiguously related to the initial value of TP, – it is implied, the rolling was carried out in the same direction as has been for the original bars. The obtained data also showed that in the texture changes of these materials, the twinning deformation systems play a supremely dominant role, and the slip activity in this is insignificant. This fully agrees with the developing concept on texture formation in hcp metals [5–7].

In the present work, the first task is to study the TP changes during the cross rolling [8] of plates from the Zr-2.5%Nb alloy used as a structural material in RBMK reactors – of rings cut from a tube of this alloy and straightened to a flat shape – rolling of them in tangential direction of the original tube. The task was mainly aimed at revealing the distinctive features of texture dynamics in comparison with the results of investigation of plates cut and rolled in longitudinal tube direction [3, 4]. Preliminary results of the investigations gave a motive to formulate another one task – studying the influence of certain features of the processing on the changes of texture of alloy plates.

MATERIAL AND TECHNIQUE

The investigations were carried out on the material of ∅15.0×1.5 mm tube of Zr-2.5%Nb. Texture measurements did on an X-ray diffractometer DRON4-07 in CuK_α-radiation with the usual Bragg-Brentano scheme.

First of all, the texture of the tube was investigated. For this, a sample was made in the form of stack of tube's segments. For the studies, three flat surfaces of the stack were prepared in accordance with three directions of the tube. This is the axial (TAD), radial (TRD) and tangential tube direction (TTD) (Fig. 1).

Designed for subsequent deforming, initial bars of the samples were also cut out of the tube in the form of rings (see Fig. 1). Then the rings were straightened to a flat shape. For maximally uniform their straightening, tubes of different diameters were used as punches.

After this, the standard finish annealing of the made plates was carried out – at 580 °C for 3 hours – until the complete separation of the β-Nb-phase. The plates were further deformed by cold rolling along their length (see Fig. 1). So, the rolling direction of the plates (RD) coincided with the TTD of the tube, the normal direction

(ND) did with the tube's TRD, the plates' transverse direction (TD) did with the tube's TAD. Thus, specimens with deformation to 7, 14, 19, 30, 39, and 55% were made. Starting from the initial state, each rolling step – from each degree to the next one – was carried out one-act. Between the steps, technological breaks were made for gauging.

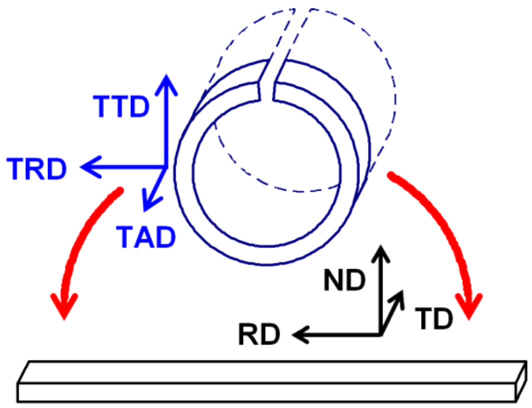

Fig. 1. Scheme of bars preparation

The investigated surfaces of the stack of original segments and surfaces of the plates were cleaned by etching in a reagent with a volume combination of water, nitric and hydrofluoric acid in the proportion of 9:5:1.5.

Texture of both the stack and the plates was investigated by the method of inverse pole figures (IPFs) [9, 10]. Investigations by this method allow most accurately determine the TP of polycrystalline materials with the hexagonal and tetragonal crystal lattice.

The essence of the method lies in the analysis of density distribution of crystallographic planes orientations in polycrystalline material along the selected direction. For hcp lattice, it is the distribution of the (*hkil*) normal to these planes (from the full (*hkil*) set of registerable X-ray reflections), which coincide with the chosen measurement directions. In this case, these are the plates' ND and the tube's TAD, TTD, TRD.

In the process of IPF building, for each pole (*hkil*), the density of the corresponding orientations – of the poles – is calculated; their regular distribution is constructed and displayed on the IPF by isolines or the color scale (see below).

For calculating pole densities in selected measurement direction (surface of sample), integral intensities of all reflections – *I*(*hkil*) values – being defined. The densities of the poles are calculated further by the following formula:

$$P_i = \frac{1}{R}\frac{I_i}{I_{0i}}; \qquad R = \sum_i A_i \frac{I_i}{I_{0i}}, \tag{1}$$

where *i* formally means (*hkil*); I_{0i} are preliminary either calculated or experimental values of X-ray intensities from such a material in its non-textured state; A_i are the statistical weights of the *i*-th reflection – the fractions of the space of the crystallographic orientations of the grains giving the *i*-th reflection [11].

Using the obtained $\{P_i\}$ set, the TP values are calculated [10, 11]:

$$f_j = \langle \cos^2 \alpha_i \rangle_j = \sum_i A_i P_{ij} \cos^2 \alpha_i , \tag{2}$$

where α_i is the angle between the basis normal of the reflecting grains (it is the "*c*" axis of the hcp cell) and the *j* direction of measurements (it is the normal to the plate plane (ND)).

In order of accounting the textural differences that exist between the sides of the plates – the outer and inner surfaces of the original tube – the measurements were carried out from both sides. In this connection the corresponding notations were taken – "outer" and "inner" sides.

To control the texture non-uniformity along the length of the plate – this circumstance is provided – the measurements were in some cases carried out from different parts of the plate surfaces. On the basis of this, the satisfactory texture uniformity along the length of the plates was established. The random error in determining the TP ($0 \le f \le 1$), with exception of several cases, is approximately ±0.005. It is meant possible to have a systematic error related to texture irregularity along the plate's thickness.

RESULTS AND DISCUSSION

Fig. 2 shows the IPFs of the original tube in three measurement directions The main crystallographic (*hkil*) directions are indicated on the IPFs and the corresponding $P_{(hkil)}$ values are given.

According to the obtained data (see Fig. 2), the tube's texture by its characteristic features is similar to the usual plates rolling texture of titanium subgroup metals, if to match the plates' RD to the tube's TTD. Thus, the "*c*" axes ((0002) normals) are basically distributed in a fan-like manner between the directions TTD and TRD of the tube. This is the result of orienting the prismatic (*hki0*) normals along the TAD. Thus, the choice of the TTD as the subsequent rolling direction corresponds with sufficient similarity to the characteristic features of the cross rolling of the plates (with 90° turn).

Fig. 3 shows the IPFs of the original and deformed (by 7%) plate. They are built by the results of measurements from both sides. According to the figure, even at 7% deformation, noticeable differences are observed in the pole figures of the plates, namely, differences along the ND between the $P_{(11\bar{2}0)}$ values and especially between $P_{(10\bar{1}0)}$.

Significant differences are also found between the results of the TP calculation for both sides of all the plates (Fig. 4). To determine the degree of reliability of these data, some TP values, as mentioned above, were determined twice: by measuring from the center and edge of the plates, and this is indicated on the graphs by pairs of dots. Despite the satisfactory reproducibility of measurements for each side, such differences still remained.

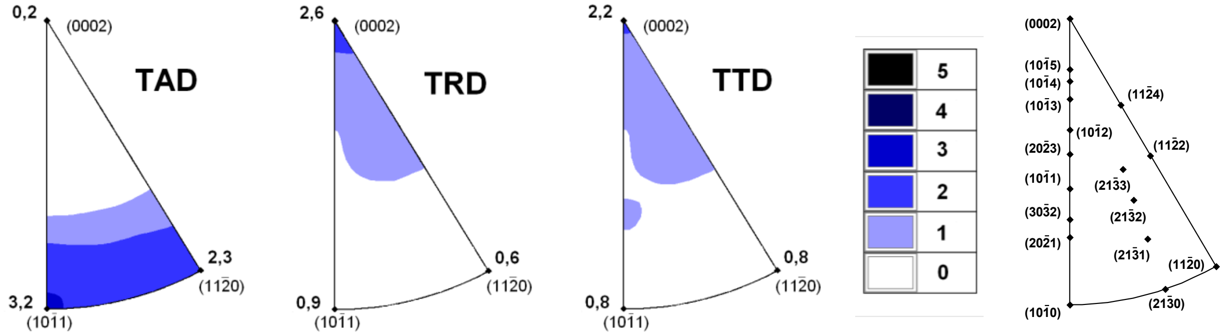

Fig. 2. IPFs in three directions of the Zr-2.5%Nb tube. On right, the dislocation of (hkil) poles is shown

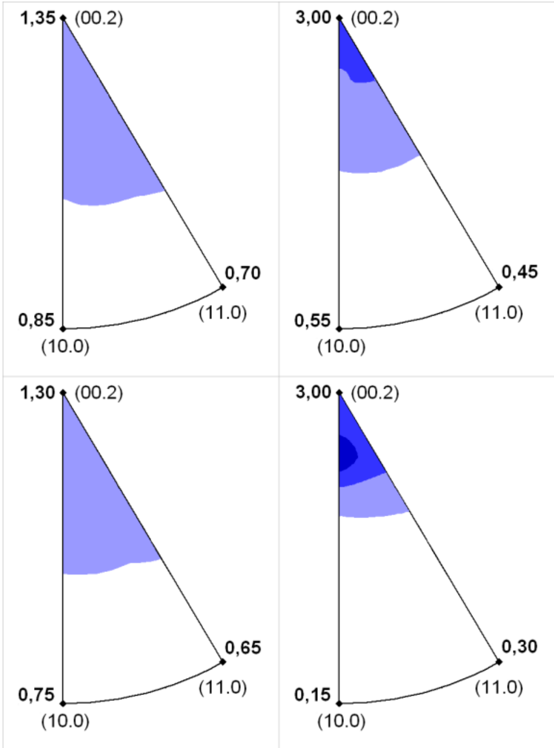

Fig. 3. IPFs of Zr-2.5%Nb plates: initial (left) and deformed 7% (on right) from "outer" (up) and "inner" side (below)

In view of this, it was taken into account that this texture ambiguity can be related to the deformation of the surfaces of the rings when they were straightened. The degree of such deformation was estimated by the tube sizes – this is the ratio of the thickness to its median diameter, which is 10%.

In accordance with this, the correction of the obtained data for the straightening effect was taken into account by shifting the points' positions on the TP plot: for the "inner" surfaces by +10% along the deformation axis, for the "outer" sides by -10%. These actions are marked by arrows (see Fig. 4). The result of such transformations is shown in Fig. 5, where the degree of deformation on the plates' surfaces – i. e. plastic deformation of plates in the usual sense plus or minus the deformation of the surface in view of preliminary alignment – is plotted along the abscissa axis.

According to Fig. 5, such acts led to a noticeable regularization of the graph. Moreover, the regularity turned out to be optimal within 0.5% error, i. e. when corrected ±(10±0.5)%, satisfactorily corresponding to the result of the amendment.

On the other hand, the synthesized graph (see Fig. 5), as can be seen, nevertheless shows a significant singularity – sharp oscillations of the TP beginning with a deformation of 4% (14% on Fig. 4).

This feature is fundamentally inconsistent with the results of similar studies of annealed pure metals – hafnium, zirconium and Zr-2.5%Nb alloy [3, 4]. In these works the regularity and even linearity of TP changes at the initial stage of deformation was observed, as well as its stationarity under deformations of about 30% and above.

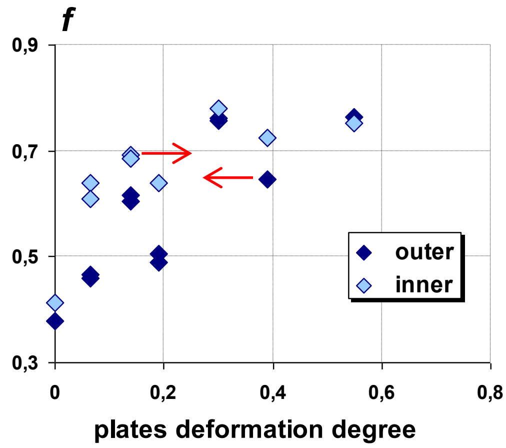

Fig. 4. Changes of TP on "outer" and "inner" sides of plates

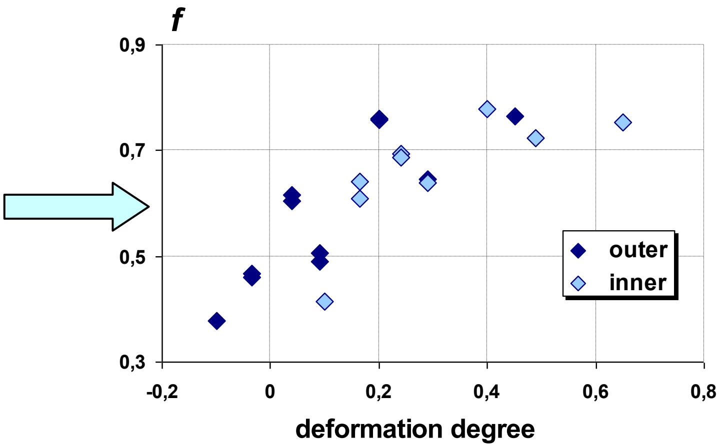

Fig. 5. Changes of TP with deformation degree on the plate surfaces

An attention is drawn to the details of this effect: beginning from the deformation of 4%, the position of the points corresponding to the “outer” sides (and the several points of the “inner” sides, Fig. 5), practically take the amplitude positions in the oscillations, i. e. in each such position, the TP dynamics changes sign. In other words, this dynamics is influenced in a special way by a certain change in the internal state of the material after current rolling step.

This is formally associated with relaxation of stresses after the rolling acts, as a result of which, obviously, the trends of reverse TP changes arise.

In the present case, conditions were in reality created for this – a time was taken between the rolling steps because of certain measurements the samples, and this interval was initially reach about 40 min and was reduced about to 5...10 min at the end of the stage of preparation of deformed samples.

This is considered as the main reason of the observed effect. A special study was carried out to verify it. Thus, on the similar deformation scheme, however, at intervals of not more than two minutes, samples from the same initial batch were obtained with the deformation of 19 and 23%. Measurements, processing of results and input of corrections were carried out by similar way. The result is shown in Fig. 6 together with the previous data (see Fig. 5) which marked by dotted line.

The obtained result confirmed the expectation that the continuity of the stepwise deformation process ensures the regularity of the TP dynamics on surfaces of the plates, considering the location of new points (see Fig. 6). Moreover, the points of the previous graph (see Fig. 5) – most of the points corresponding to the “outer” sides, what is indicated in Fig. 6 by small dots, – are also embedded in the regularity of the assumed graph. As a result, the TP dependence (dashed line) is seen, formally similar to that obtained in [4]: there is the initial stage of growth with signs of its linearity – in present case with the rate $df/d\varepsilon \approx 2$ – and also the subsequent stationary stage with $f \approx 0.75$ (see Fig. 6).

On the other hand, it can be seen from Figure 6 that the elimination of the deforming is manifested in the sharp falls of the TP values (see Fig. 5) relatively to the corrected version, beginning from the deformation of 4% (14% for a whole plate, Fig. 4). Such falls involve most points corresponding to the internal sides of the rings, what indicates the greatest texture instability on the “inner” surfaces.

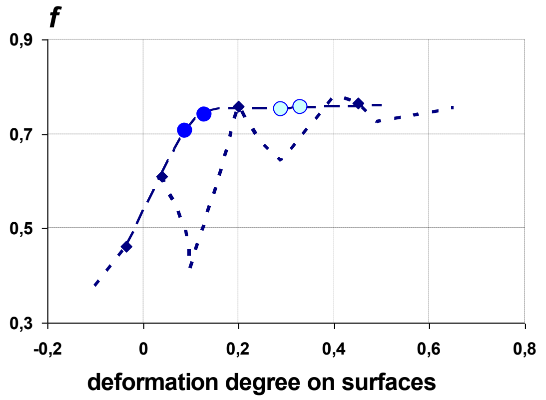

Fig. 6. The point positions on graph of TP values for “outer” (●) and “inner” (○) plates surfaces at deformation without delays

In view of this, the next stage of research is to investigate near-surface regularity of the texture parameter. Thence, a study was made of the TP changes within 30 µm of the etching depth of the plates. The subject of the study was a plate made of such tube and annealed at 580 °C for 24 hours. The results are shown in Fig. 7,a.

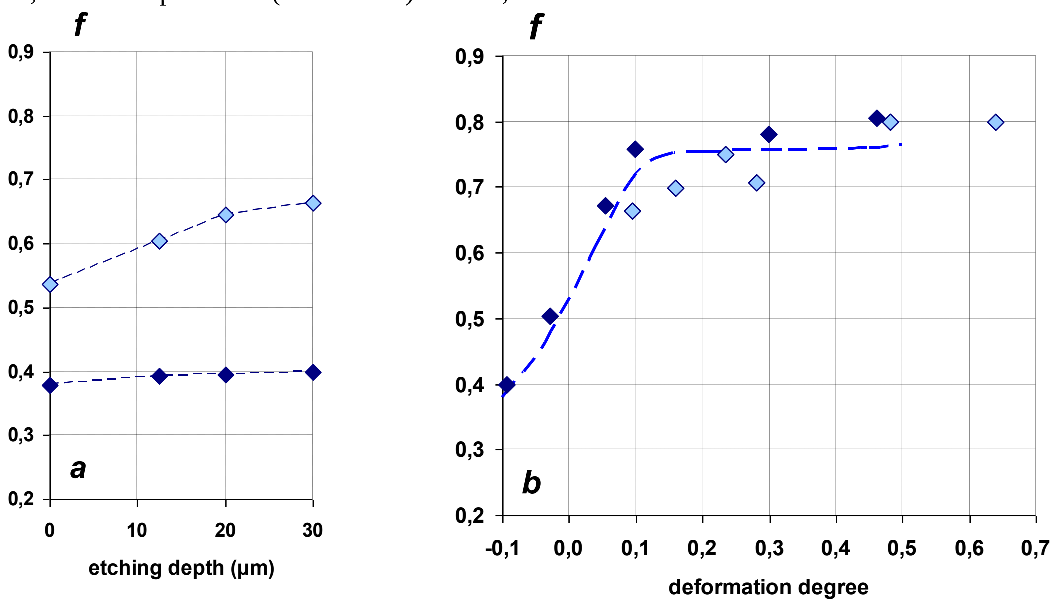

Fig. 7. TP changes on “outer” (◆) and “inner” (◆) surfaces of Zr-2.5%Nb plates: a – with etching depth of original plate; b – with deformation degree of them

According to Fig. 7,a, the TP on the “inner” side of the original plate undergoes significant changes after the acts of etching. In particular, it linearly changes up to a depth of 20 µm. At the same time, on the “outer” side, starting at 12 µm depth, the TP becomes practically unchanged, showing the trend of linear changes between its values on the surfaces. This gives grounds to consider its value on middle of each plate to be true average value for it.

After this, the TP was re-measured after etching all the samples to a depth of more than 30 μm. The results are shown in Fig. 7,b in conjunction with the corrected graph (see Fig. 6) marked by dashed line.

According to the data presented (see Fig. 7,b), TPs on the “inner” sides of the plates showed irregularity, which reflected their pre-history (see Fig. 5). At the same time, as expected, the data from the “outer” sides showed their regularity and similarity with the corrected graph of Fig. 6, exceeding its data as a whole by the value 0.03: the rate of change in the TP at the initial stage is 1.8...1.9; the values of TP at the stationary stage are 0.785±0.015. Despite the residual irregularity of the TP on the “inner” sides, the last graph of the TP on the “outer” sides should be considered the true graph of TPs for textural-uniform plates of this alloy.

It also follows from the data given that the relaxation of stresses in the plates, occurring for few tens of minutes, focuses on their surfaces, and to a greater extent – on the “inner” side. The clear manifestation of this effect in these studies gives grounds to presume that a twinning can be a structural mechanism which creates returning TP changes in a discontinuous process of deformation with the rate of ~ 4 s^{-1}.

CONCLUSIONS

Using the X-ray method of inverse pole figures (IPFs), texture of cold deformation by rolling plates of Zr-2.5%Nb alloy has been investigated. The plates were manufactured in the following way:

– the bars in the form of rings were cut from the ∅15.0×1.5 mm tube and straightened up to a flat shape;

– the resulting plates were annealed at a temperature of 580 °C for 3 hours – until the complete segregation of the β-Nb-phase and creating the partial recrystallization state;

– the plates were rolled along the length of the original rings up to 7, 14, 19, 30, 39, and 55% doing one rolling act from each degree to next one and with technological intervals of 15...40 min between the rolling acts.

IPFs were built and the values of the Kearns’ TP for both sides of the plates were calculated.

Significant differences in texture and TP on the surfaces of different sides of the plates are noted, which is associated with deformations caused by straightening the original rings.

An optimal graph of the changes in the TP with the degree of deformation of the plate surfaces was obtained by input to the plates’ deformation degrees an additive correction which due to the straightening of the rings and calculated from their geometric parameters. An irregularity of the built graph in the form of sharp oscillations of the TP values, beginning from ~ 15% deformation of the plates, is revealed.

It has been established that such oscillations are associated with accumulation of residual stresses and their relaxation on plate surfaces in a few tens of minutes after deformation, which is accompanied by incipience of trends of return TP changes. A hypothesis is made that twinning is a mechanism of such relaxation.

After eliminating the surface stress-relaxation effects, a graph of the TP changes with degree of cross rolling of Zr-2.5%Nb plates was obtained for case of uniformity of initial texture along their thickness. A clearly expressed staged nature of the graph is revealed – existence of an initial stage of almost linear growth of TP and the subsequent its stationary stage. Numerical estimates of the parameters of these stages are made.

The results of these studies broaden the possibilities of predicting, based on the initial texture, deformation changes of texture parameter of zirconium-based alloys.

Existence in principle of the structural effect of stress relaxation in materials can lead to inaccuracies in control of texture characteristics. It is considered advisable to take this into account in cases of high speeds of deformation, irregularities of it and its process, low temperatures of processing.

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ВЛИЯНИЕ ПОПЕРЕЧНОЙ ПРОКАТКИ И ЭФФЕКТА РЕЛАКСАЦИИ НАПРЯЖЕНИЙ НА ТЕКСТУРУ ПЛАСТИН СПЛАВА Zr-2.5%Nb

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Методом обратных полюсных фигур (ОПФ) проведены рентгеновские исследования влияния холодной прокатки на текстуру и текстурный параметр Кернса (ТП) пластин, изготовленных из колец трубы ∅15,0×1,5 мм сплава Zr-2.5%Nb, выпрямленных до плоской формы. Прокатка осуществлялась в направлении, перпендикулярном направлению продольной прокатки исходной трубы, с деформацией от 7 до 55% и с интервалами ~15…40 мин между проходами. Построены ОПФ и график зависимости ТП от деформации на поверхностях пластин. Отмечены резкие осцилляции графика. Исследованием образцов, протравленных на 30 мкм, а также исходных образцов, выборочно изготовленных без задержек в процессе прокатки, установлено, что это связано с поверхностной релаксацией напряжений в течение 15…40 мин. Определен график изменений ТП для условий однородности исходной текстуры сплава и непрерывности процесса прокатки. Выявлен стадийный характер зависимости ТП от деформации – наличие начальной стадии практически линейного роста и последующей стадии незначительных изменений.

ВПЛИВ ПОПЕРЕЧНОЇ ПРОКАТКИ І ЕФЕКТУ РЕЛАКСАЦІЇ НАПРУГ НА ТЕКСТУРУ ПЛАСТИН СПЛАВУ Zr-2.5%Nb

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Методом зворотних полюсних фігур (ЗПФ) проведені рентгенівські дослідження впливу холодної прокатки на текстуру і текстурний параметр Кернса (ТП) пластинів, виготовлених з кілець труби ∅15,0x1,5 мм сплаву Zr-2,5%Nb, випрямлених до плоскої форми. Прокатка здійснювалася в напрямку, перпендикулярному напрямку подовжньої прокатки початкової труби, з деформацією від 7 до 55% і інтервалами ~ 15…40 хв, між проходами. Побудовані ЗПФ і графік залежності ТП від деформації на поверхнях пластин. Відмічені різкі осциляції графіку. Дослідженням зразків, протравлених на 30 мкм, а також початкових зразків, вибірково виготовлених без затримок в процесі прокатки, установлено, що це пов'язано з поверхневою релаксацією напруги впродовж 15…40 хв. Визначений графік змін ТП для умов однорідності початкової текстури сплаву і безперервності процесу прокатки. Виявлений стадійний характер залежності ТП від деформації – наявність початкової стадії практично лінійного зростання і подальшої стадії незначних змін.