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Effect of structure and phase composition on the physical and mechanical properties of hot extruded titanium alloy Ti–3Al–2.5V tubes

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Abstract: This study investigates the impact the hot extrusion process variables on the physical and mechanical properties of Ti–3Al–2.5V alloy tubes. The research examines four tube segments extracted from various hot-extruded tubes of Ti–3Al–2.5V alloy, with an outer diameter (OD) of 90 mm and a wall thickness of 20 mm. The manufacturing process involves expanding sleeves with a horizontal hydraulic press to achieve an OD of 195 mm, followed by heating to 850–865 °C prior to extrusion. The tube segments are labeled as *1*, *2*, *3*, and *4*, corresponding to their order of production. Our findings demonstrate that an increase in the number of extrusions in the $\alpha + \beta$ area from tube 1 to tube 4 leads to a reduction in the primary α-phase volume fraction and an increase in the β-transformed structure volume fraction. These changes are attributed to the higher final extrusion temperature resulting from more intense deformation heating during hot tooling (die and mandrel) processes. Additionally, elevating the final extrusion temperature from tube *1* to tube *4* leads to a notable decrease in the residual β-solid solution volume fraction and a reduction in the "sharpness" of the α-phase tangent-oriented texture. The alterations in the structural and phase state of the alloy from tube *1* to tube *4* are found to influence the contact modulus of elasticity and microhardness. These identified relationships can be utilized to optimize the process variables for the extrusion of multiple Ti–3Al–2.5V alloy tubes.

Keywords: titanium alloy Ti–3Al–2.5V, hot extrusion, structure, texture, mechanical properties.

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Влияние структурно-фазового состояния на физико-механические свойства горячепрессованных труб из титанового сплава Ti–3Al–2,5V

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Аннотация: Исследовано влияние изменения параметров горячего прессования на физико-механические свойства труб из сплава Ti–3Al–2,5V. Материалом для исследования служили четыре патрубка, отобранные от разных горячепрессованных труб из сплава Ti–3Al–2,5V с внешним диаметром 90 мм и толщиной стенки 20 мм, полученных из экспандированных гильз с внешним диаметром 195 мм на горизонтальном гидравлическом прессе. Экспандированные гильзы перед прессованием нагревались до температуры 850–865 °С. Образцам исследуемых горячепрессованных труб присвоены номера *1*, *2*, *3* и *4* согласно последовательности их получения в промышленных условиях. Показано, что увеличение количества проведенных прессовок в $\alpha + \beta$-области от трубы *1* к трубе *4* приводит к закономерному уменьшению объемной доли первичной α-фазы в их структуре, а также к росту объемной доли β-превращенной структуры вследствие повышения температуры окончания прессования, вызванного более активным деформационным разогревом из-за увеличения температуры инструмента (матрицы и иглы). Обнаружено, что фиксируемое структурно повышение температуры окончания прессования от 1-й трубы к 4-й влечет за собой характерное уменьшение объемной доли остаточного β-твердого раствора и снижение «остроты» наблюдаемой тангенциальной текстуры α-фазы. Установлено, что выявленные изменения структурно-фазового состояния сплава от 1-й трубы к 4-й оказывают закономерное влияние на получаемый в них уровень свойств – контактного модуля упругости и микротвердости. Полученные закономерности необходимо учитывать при разработке технологического режима многоразового прессования труб из сплава Ti–3Al–2,5V.

Ключевые слова: титановый сплав Ti–3Al–2,5V, горячее прессование, структура, текстура, механические свойства.

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Introduction

Pseudo-α-titanium alloys exhibit a distinctive combination of advantageous properties, such as high specific strength, corrosion resistance, and excellent manufacturability. They find application in the production of critical components, including tubes [1–4]. One such alloy is the Ti–3Al–2.5V pseudo-α-titanium alloy which conforms to the ASTM B338 Standard Specification for Seamless and Welded Titanium and Titanium Alloy Tubes for Condensers and Heat Exchangers. This alloy is widely employed in the manufacturing of tubes [5; 6], and its manufacturability allows for the production both hot-extruded [7] and cold-drawn tubes [8; 9].

Nikol’skii L. et al. [10], Kosmatskiy Ya. et al. [11] have observed that the hot extrusion process used to fabricate tubes and other products from the Ti—3Al—2.5V alloy can exhibit temperature and deformation variations. These variations arise due to deformation heating of the workpiece and tooling, as well as potential cooling of the product surfaces when they come into contact with a colder tool.

Kosmatskiy Ya. et al. [12], Tarin P. et al. [13], and Illarionov A. et al. [14] conducted research on the Grade 9 alloy and found that temperature fluctuations during hot drawing in the α + β two-phase region influence the ratio of α- and β-phases, thermophysical properties, and deformation forces. Additionally, Pyshmintsev I. et al. [7] reported that such temperature variations impact the final structure, phase composition, and texture after cooling. Consequently, these variations are likely to affect the physical and mechanical properties of the end product.

Despite the existing research on the topic, there is a notable absence of studies investigating the effects of the structure and phase composition formed under non-stationary hot extrusion conditions on the properties of Ti—3Al—2.5V alloy tubes. Therefore, the purpose of this study is to address this research gap and explore the specific impact of non-stationary hot extrusion conditions on the properties of the Ti—3Al—2.5V alloy tubes.

Materials and methods

The samples consisted of four tube segments extracted from various hot-extruded Ti—3Al—2.5V alloy tubes, with an outer diameter (OD) of 90 and a wall thickness of 20 mm. These tubes were manufactured by utilizing 195 mm outer diameter expanded sleeves and a horizontal hydraulic press. Prior to the extrusion process, the expanded sleeves were heated to 850—865 °C. The specific extrusion process variables, including temperature and strain rate, were detailed in a previous work by the authors [15]. The four samples of the hot-extruded tubes were numbered *1*, *2*, *3*, and *4* according to the sequence of their manufacturing.

In this study, three main analytical techniques were employed to characterize the samples. Optical microscopy, *X*-ray diffraction (XRD), and micro-indentation were utilized to measure the Vickers hardness and contact modulus of elasticity of the tubes. The microstructure of the tubes was analyzed using a GX51 microscope (Olympus, Japan). For sample preparation, micro slides were etched using an aqueous solution consisting of a mixture of hydrofluoric and nitric acids (1 part HF + + 3 parts HNO_3 + 5 parts H_2O) as suggested by Anoshkin N. et al. in [16], following the method suggested by Anoshkin N. et al. in [16]. XRD analysis was performed on a Bruker D8 Advance *X*-ray diffraction platform (Bruker, Germany) using copper CuK_α radiation in the $2\theta = 34°\div102°$ range. The XRD data were analyzed using Rietveld refinement [17] with the TOPAS® 4.2 software package.

Microgeometry and contact modulus of elasticity measurements were carried out using the Oliver-Farr micro indentation hardness test [18]. A MHTX micro indenter (CSM Instruments, Switzerland) was utilized, applying a 9 N load with 6 measurements conducted per sample.

Results and discussion

In order to assess the phase state of the tubes, *X*-ray diffraction (XRD) analysis was conducted on the longitudinal sections of the samples (Fig. 1).

The Rietveld refinement of the XRD patterns (Fig. 2) yielded the identification of two distinct phases, namely, α-phase and β-phase lines. The lattice parameters of both the α + β- and β-phases were estimated from the XRD patterns, and the corresponding volume fraction of the β-phase was determined. The results are summarized in the table below.

The olume fraction of the β-phase in all the samples varied between 4.8 to 6.2 %. There is a noticeable correlation between changes in the volume fraction of the β-phase and the lattice period; the period slightly increases with the volume fraction. This outcome is in line with expectations, as the presence of β-stabilizers (vanadium and iron impurities) in the β-solid solution

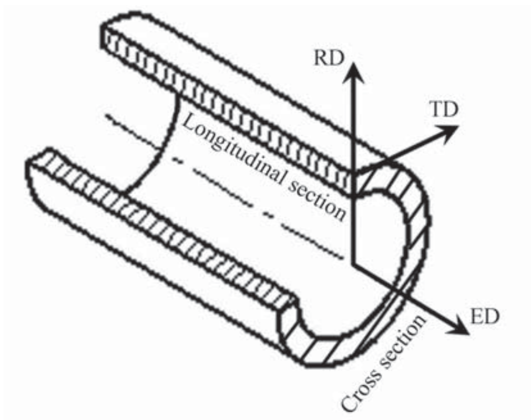

Fig. 1. The three directions are denoted as follows
RD – radial direction, TD – tangential direction,
ED – extrusion direction

Рис. 1. Эскиз трубы с указанием трех основных направлений, связанных с внешним воздействием
РН – радиальное направление, ТН – тангенциальное направление, НП – направление прессования

Lattice periods of the α- and β-phases, the volume fraction of the β-phase in hot-extruded tube samples *1—4*

Периоды решеток α- и β-фаз, объемная доля β-фазы в образцах *1—4* горячепрессованных труб

Sample No.	α-phase			β-phase	
	Lattice parameter, nm		*c*/*a*	Lattice period, nm	Volume fraction, %
	a	*c*			
1	0.29370	0.46724	1.5909	0.32242	6.2
2	0.29408	0.46770	1.5904	0.32222	5.4
3	0.29388	0.46757	1.5911	0.32202	5.0
4	0.29391	0.46761	1.5910	0.32196	4.8

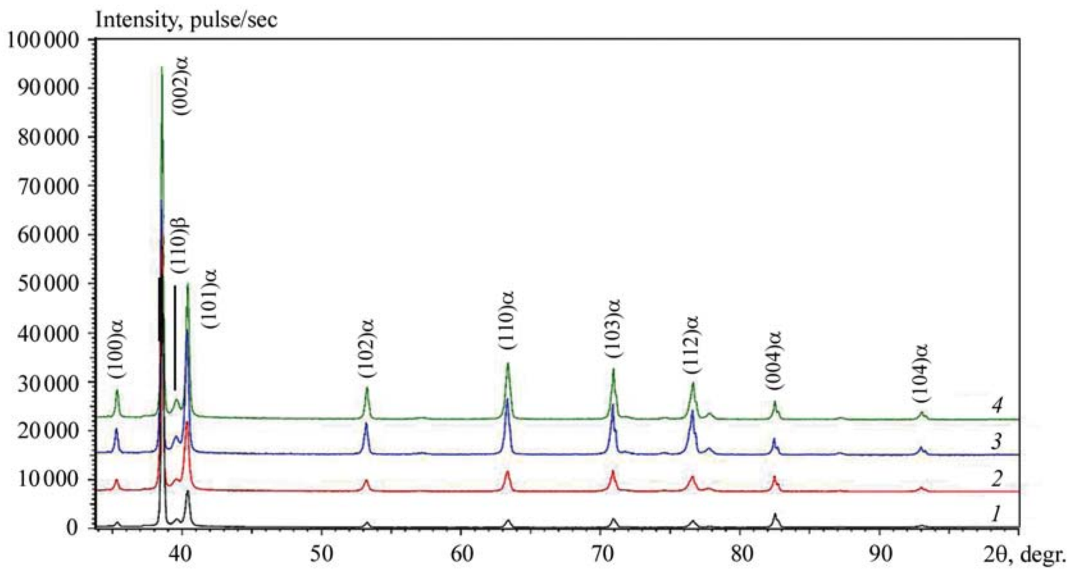

Fig. 2. XRD patterns of the hot-extruded tubes ***1—4***
Shooting in the tangential direction

Рис. 2. Дифрактограммы образцов ***1—4*** горячепрессованных труб
Съемка в тангенциальном направлении

decreases. Prior research [19—21] has established that these stabilizers contribute to the reduction of the lattice period.

Regarding the tube samples (labeled as 1-4), the c/a parameter of the α-phase falls within the range of 1.5904 to 1.5911 (refer to the table). This value is lower than the c/a values for the original sleeves, which range from 1.5913 to 1.5915, and these sleeves were expanded at a temperature similar to that of the hot extrusion process. This discrepancy suggests that the diffusion processes in the α-phase, which occur during cooling down from the extrusion temperature, are less complete compared to those in the the phase formed during cooling after expansion. The reason behind this observation is that the wall of the hot-extruded tubes is 72 % thinner than that of the expanded sleeves, leading to higher cooling rates and consequently reducing the diffusion period.

Fedulov V. et al. [22] conducted a study on the VT23 titanium alloy, which shares a similar temperature of polymorphic $\alpha + \beta \rightarrow \beta$ transformation with the Ti—3Al—2.5V alloy [1]. In their research, it was demonstrated that when the wall thickness is reduced by 65—75 % during cooling from 850 °C (close to the tube extrusion temperature), the cooling rate along the cross-section more than doubles.

An analysis of the *X*-ray diffraction (XRD) patterns of the tubes allowed for a comparison of the intensities of the α-phase lines. It was observed that the (002)α line exhibits the highest intensity in the hot-extruded tube samples *1—4*. Interestingly, for the sleeves after expansion, the (101)α line, not the (002)α line, displayed the highest intensity. This discrepancy indicates that the hot extrusion process creates a tangent-oriented basic texture of the α-phase (Fig. 3) in the two-phase region of the tube samples *1—4*. In other words, the normal to the basis plane (0001) in the α-phase grains is predominantly oriented in the tangential direction. This finding aligns with the work of Forney C. et al. [23], who reported that reduction drawing, as in our case, facilitates the formation of a tangent-oriented basic texture. It should be noted that the quality of the texture varies from tube to tube, as evidenced by the changes in the relative intensities of the primary α-phase lines in the XRD patterns relative to the (002)α line (Fig. 4).

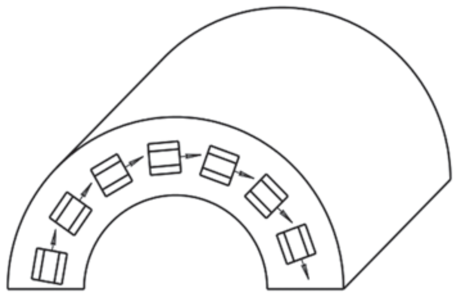

Fig. 3. Characteristic arrangement of the α-phase hexagonal cell as the tangent-oriented texture is formed in the Ti–3Al–2.5V alloy

Рис. 3. Характерное расположение гексагональной призмы α-фазы при формировании тангенциальной текстуры в сплаве Ti–3Al–2,5V

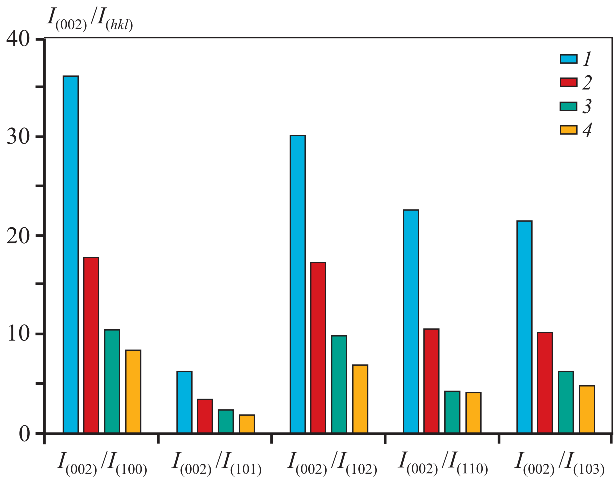

Fig. 4. $I_{(002)}/I_{(hkl)}$ ratio variations at the max intensity of the α-phase lines with different (*hkl*) indices ***1—4***

Рис. 4. Изменение отношения $I_{(002)}/I_{(hkl)}$ для максимальной интенсивности линий α-фазы с различными индексами (*hkl*) на дифрактограммах для горячепрессованных труб ***1—4***

As evident from Fig. 4 and the table, there exists a correlation between the quality of the tangent-oriented texture and the volume fraction of the β-phase: the higher the fraction, the more intense the line becomes (reflected by the higher $I_{(002)}/I_{(hkl)}$ ratio). This observation leads to the conclusion that the XRD pattern analysis for the β-phase volume fraction indicates the resistance to decomposition during cooling of the high-temperature β-solid solution. Moreover, this resistance is linked to the relationship between the condition of the α-phase texture and the physical and mechanical properties (as depicted in Fig. 5). Specifically, as the volume fraction of the β-phase decreases, both the contact modulus of elasticity and microhardness exhibit a corresponding decrease.

The observed relationship between the values can be explained as follows. Samples *1* and *2* retain a larger amount of β-phase, indicating less complete decomposition of the high-temperature β-solid solution and α-phase separation during cooling, in comparison to samples *3* and *4*. The less complete decomposition of the high-temperature β-phase usually occurs when the initial β-solid solution contains more β-stabilizers

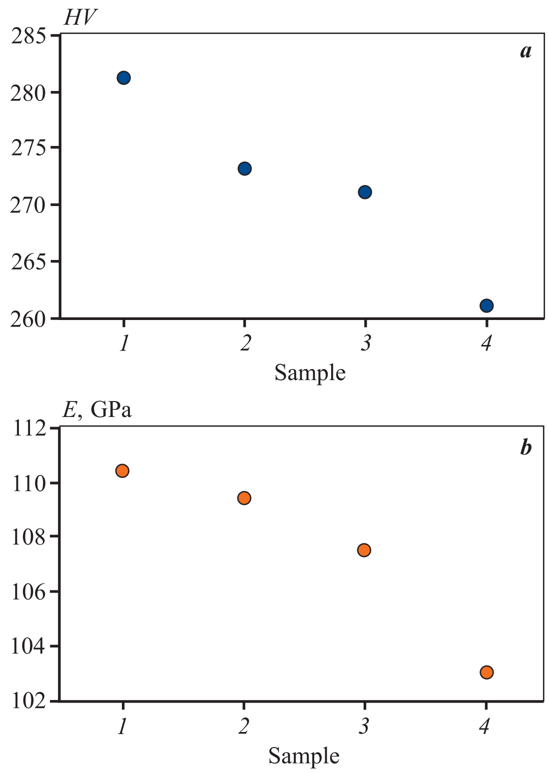

Fig. 5. Variations of the average microhardness (***a***) and contact modulus of elasticity (***b***) in samples ***1—4***

Рис. 5. Изменение средних значений микротвердости (***a***) и контактного модуля упругости (***b***) в образцах ***1—4*** горячепрессованных труб

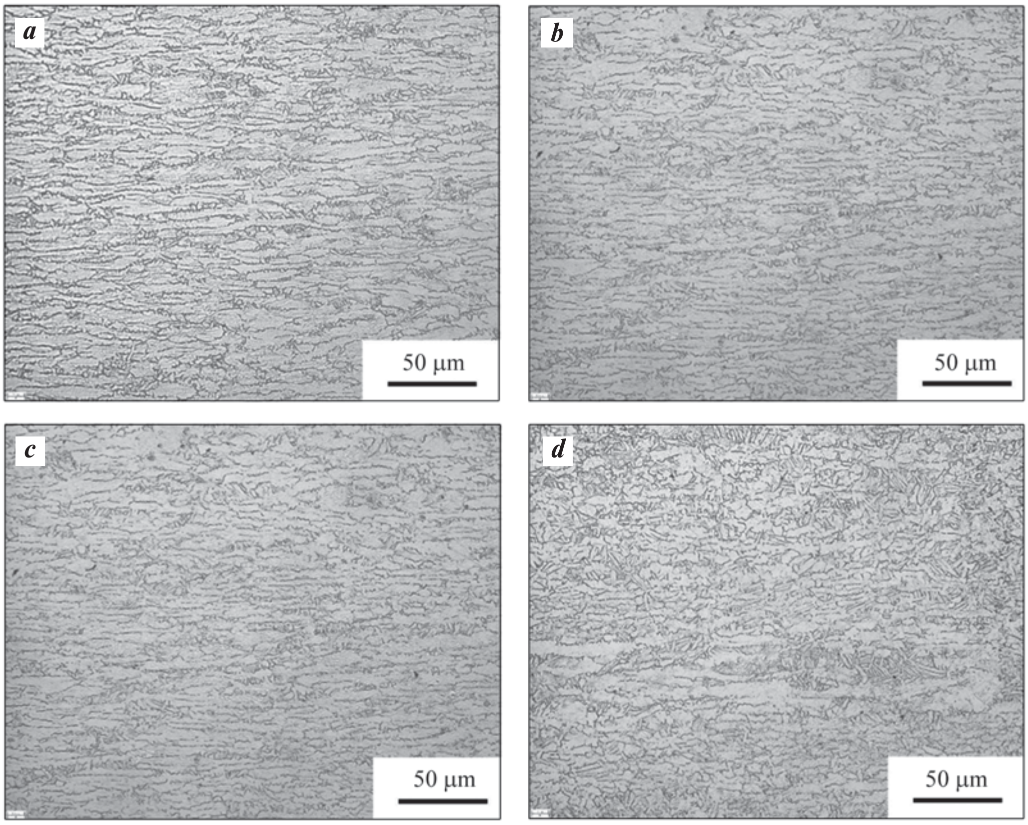

Fig. 6. Predominant longitudinal section microstructure of the ***1*** (***a***), ***2*** (***b***), ***3*** (***c***) and ***4*** (***d***) hot-extruded tube samples, Ti–3Al–2.5V alloy

Рис. 6. Преобладающая микроструктура в продольном сечении горячепрессованных труб ***1*** (***a***), ***2*** (***b***), ***3*** (***c***) и ***4*** (***d***) из сплава Ti–3Al–2,5V

before cooling. The presence of more β-stabilizers in the initial state is a result of a lower initial cooling temperature of the tube compared to the other tubes. This effect arises from the lesser deformation heating of the first Ti—3Al—2.5V alloy tubes manufactured by hot extrusion. Subsequent extrusions cause additional heating of the tools (die and mandrel), leading to reduced heat removal from the tube to the tool. Similar effects have been observed in other metal extrusion processes [24; 25].

The proposed explanation is further supported by the microstructural analysis of the tubes. A thorough examination of typical structures found in most areas of the longitudinal tube section revealed that samples *1* and *2* (Fig. 6, *a*, *b*) are dominated by primary α-phase grains elongated along the direction of extrusion. Small regions of the β-transformed structure exist between the grains, appearing as clusters of secondary α-phase thin plates with different orientations and β-phase interlayers. The formation of the β-transformed structure occurs as a consequence of the high-temperature β-solid solution decomposition during cooling from the extrusion temperature.

A notable characteristic of the structure in samples *3* and *4* (Fig. 6, *c*, *d*) compared to samples *1* and *2* is a significant decrease in the volume fraction of the primary α-phase elongated along the direction of extrusion. This reduction results in an increase in the volume fraction of the β-transformed structure areas. The primary α-phase exhibits partial fragmentation and spheroidization, indicating dynamic recovery processes [26]. Furthermore, the β-transformed structure areas become broader, and the secondary α-plates are enlarged.

The observed difference in the structure of the tube samples can be logically explained by the gradual increase in the end-extrusion temperature from sample *1* to sample *4*. This temperature rise occurs due to higher deformation heating caused by the increasing temperature of the die and mandrel during the extrusion process of tubes *1* to *4*. Consequently, this intensifies relaxation recovery and dissolution of the primary α-phase, leading to an increase in the volume fraction and size of the high-temperature β-phase areas during the extrusion process. Upon cooling, the β-phase decomposes in these areas, forming plates of the secondary α-phase.

The observed decrease in microhardness from sample *1* to sample *4* (see Fig. 5) can be attributed to the more intense recovery processes in the primary α-phase, which are associated with the removal of deformation hardening, and the enlargement of decomposition products in the β-transformed matrix. This conclusion is further supported by comparing the β-phase volume fraction with the thermodynamic analysis conducted in the ThermoCalc software [14].

The increase in extrusion temperature in the two-phase α + β region leads to a higher amount of β-solid solution and a depletion of β-stabilizers (vanadium and iron). This, in turn, reduces the resistance of the β-phase to decomposition during subsequent cooling. As a result, in samples *3* and *4*, which were heated to higher temperature during extrusion compared to samples *1* and *2*, the decomposition of the β-solid solution starts at higher temperatures and leads to the formation of larger secondary α-phase plates. Additionally, the decomposition process is more complete, resulting in a smaller volume fraction of the residual β-solid solution (as indicated in the table).

Unlike the primary phase, the secondary α-phase plates were not deformed during extrusion. Therefore, they lack a clear orientation or pronounced texture. The XRD patterns demonstrate a decrease in the intensity of the (002)α lines from sample 1 to sample 4 relative to the intensity of other α-phase lines. This indicates a decrease in the sharpness of the tangent-oriented prism texture. Consequently, there is a slight decrease in the contact modulus of elasticity (E), measured in the tangential direction, from sample 1 to sample 4. This is consistent with the well-known fact that the α-phase exhibits the maximum E value along the <001> direction [27].

In our view, the dominant high-modulus orientation <001> in the direction of the contact modulus of elasticity measurements results in average modulus values (ranging from 103 to 110 GPa) that are close to or even above the upper values typical for Ti—3Al—2.5V alloy products (ranging from 95—105 GPa) [2].

Conclusions

1. The study demonstrates that an increase in the number of α + β-area extrusions, from tube *1* to tube *4*, leads to a reduction in the volume fraction of the primary α-phase and an increase in the volume fraction of the β-transformed structure due to end-of-extrusion temperature rise caused by more intense deformation heating of the die and mandrel.

2. Furthermore, the investigation reveals that the elevation of the final extrusion temperature from tube *1* to tube *4* results in a distinctive decrease in the volume fraction of the residual β-solid solution and a reduction in the “sharpness” of the α-phase tangential texture.

3. Additionally, it was observed that the aforementioned changes in the structure and phase state, from sample *1* to sample *4*, have an impact on the contact modulus of elasticity and microhardness.

4. These established relationships provide valuable insights for adjusting the process variables in the multiple Ti—3Al—2.5V alloy tube extrusion.

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