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FINAL REPORT ON THE START PROGRAMME

The effect of laser shock peening on crystallographic texture evolution of VT1-0 and VT6 titanium alloys

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LIST OF ABBREVIATIONS AND SYMBOLS

ODF – orientation distribution function
CS – coordinate system
HCP – hexagonal unit cell
InPF – incomplete pole figure
PF – complete pole figure
MTEX – set of tools in Matlab package for processing of experimental pole figures
LSP – laser shock peening
RD - rolling direction
ND - normal direction
EBSD - electron back scattering diffraction
ASCII - American Standard Code for Information Interchange

CONTENTS

PURPOSE AND OBJECTIVES

The purpose of work was study of crystallographic texture of titanium alloys VT1-0 and VT6 before and after laser shock peening (LSP) using X-ray diffraction. Titanium alloys are important materials in various industries, including the aircraft and space industries, due to their high strength, lightness and corrosion resistance. The changes in the crystallographic structure of these materials can have a significant impact on their mechanical properties, including strength, plasticity, and fatigue properties.

To achieve the stated objective within the framework of the scientific research, the following tasks were set and addressed:

1. Study of changes in the crystallographic texture titanium alloys before and after laser shock peening (LSP), measured using the PANalytical Empyrean X-ray diffractometer.
2. Investigation of homogeneity crystallographic texture in different regions of the rolled sheet.

The obtained results will deepen the understanding of the processes occurring in titanium alloys during laser treatment, as well as contribute to the optimization of processing technologies aimed at enhancing their mechanical properties.

1. INTRODUCTION

Alloys VT1-0 and VT6 are used as structural materials for manufacturing various types of parts and structures. They are utilized in the aerospace industry for producing assembled structures of aircraft (aviation compressor blades of gas turbine engines), semi-finished products, and pressure vessels.

Improving the durability of structural materials is a crucial task, especially in the aviation industry. There are many methods of surface treatment for alloys to enhance their physical and mechanical properties. Processing techniques such as ultrasonic [1] or shot peening are used in the industry to strengthen the surface layer of a product. However, these methods do not allow for the processing of complex-shaped parts, unlike the LSP method [2].

The process LSP involves irradiating the surface of the target material with short (on the order of nanoseconds) laser pulses. The general scheme of LSP process is shown in Figure 1.

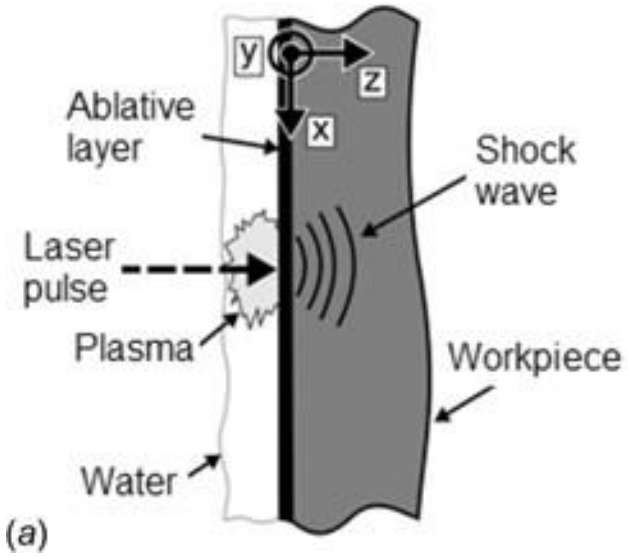


Fig.1. Schematic diagram of material processing with LSP

The surface of the target is usually pre-coated with an ablative layer, such as black paint or aluminum tape, to increase the absorption of laser energy and avoid thermal damage to the surface. The laser vaporizes the consumable layer, and the resulting vapor absorbs the laser energy. Continuous absorption of laser energy leads to the ionization of atoms into a rapidly expanding plasma. The expansion of the plasma between the target surface and the consumable layer generates short-duration, high-intensity pressure pulses. Part of this energy propagates through the target material in the form of a shock wave, which is how the processing gets its name. The pressure pulse acts on the area being processed and creates compression in the direction of the shock wave's propagation. As the shock wave travels through the material, plastic deformation occurs. This plastic deformation leads to changes in the microstructure of the sub-surface layers of the material.

The authors of work [3] demonstrated that after conducting of laser shock peening (LSP) on alloys VT6 and VT1-0, no changes were observed in the microstructure (grain size, mechanical texture). It was shown that under certain processing conditions, there is an increase in the fatigue characteristics of the material.

It is well known that crystallographic texture significantly affects the properties of polycrystalline materials. For example, the sharp texture of magnesium leads to such

anisotropy in properties that magnesium alloy sheets, after rolling, hinder the stamping process [8]. It is known that transformer steel has high magnetization coefficients when its texture is close to (110)[001] (Goss texture) [9]. By controlling and altering the texture during certain technological processes, it is possible to modify the specified properties of the material.

There have been few studies on the influence of laser shock peening (LSP) on the crystallographic texture of alloys. This work is dedicated to the issue of the impact of LSP on the texture of titanium alloys VT1-0 and VT6.

2. QUANTITATIVE TEXTURE DESCRIPTION

Polycrystalline materials consist of crystallites which orientation distribution largely determines the macroscopic properties of the whole sample. During deformation, crystallites can experience rotations that result in different orientation distributions, i.e., the appearance of one or another crystallographic texture. Crystallographic texture analysis has an important role in materials science related to the description of the relationship between the structure and properties of solid matter.

Crystallographic texture is a distribution of grain orientations in a polycrystalline sample [6]. In order to assign the orientation g of the crystal in the sample, it is necessary to introduce two coordinate systems: the sample coordinate system (external coordinate system) and the crystal coordinate system. The external coordinate system is chosen based on the symmetry of the sample or according to the geometry of the processing.

The coordinate system of the crystal is defined by the directions within the crystal (depending on the symmetry of the crystal). The orientation g is introduced as the rotation of the sample coordinate system relative to the crystal coordinate system:

$$C_c = g \cdot C_s$$

, where $C_s и\ C_c$ – the coordinate systems of the sample and the crystal, respectively. In texture analysis, Euler angles are used to represent the orientation $g = \{\alpha, \beta, \gamma\}$.

The rotation is set:

1. By rotating the coordinate system of the sample by an angle α around the z-axis.
2. By rotating the coordinate system of the sample by an angle β around the y'.
3. By rotating the coordinate system of the sample by an angle γ around the z''.

To describe the crystallographic texture of materials, the orientation distribution function (ODF) is used. By knowing the ODF and the properties of single crystals, one can calculate the properties inherent to the polycrystalline state. The orientation distribution function (ODF) is a probability density. The ODF $f(g)$ (the relative volume of crystals with a given orientation) is calculated using the equation:

$$f(g) = \frac{dV(g)/V}{dg}$$

, where $dV(g)/V$ – relative volume of crystals and g – orientation.

The orientation distribution function (ODF) is defined in such a way as to possess the property of normalization:

$$\int f(g)dg = 8\pi^2$$

The orientation distribution function (ODF), depending on the symmetry of the sample and the material being studied, may possess certain symmetry properties.

The orientation distribution function (ODF) is traditionally calculated from pole figures measured using X-ray or neutron diffraction. For cubic or hexagonal cells, from 2 to 4 pole figures are required for the calculation of the ODF. An angular resolution of 5° is usually sufficient to describe the most typical textures of deformed or recrystallized metals and alloys.

Pole figures are represented by stereographic projections of a specific set of crystallographic planes for all crystallites (grains) of a given polycrystal. The position of the points on the pole figure is determined using radial and azimuthal angles.

The analytical representation of PF is given by equation:

$$P_h(\boldsymbol{y}) = \frac{1}{2\pi}\int f(\{h,\varphi\}^{-1}\{y,0\})d\varphi$$

The orientation distribution function (ODF) is calculated if the experimental pole figures are known. This task is fundamental in quantitative texture analysis. Several methods exist for ODF reconstructing from measured PFs. One of these methods is the harmonic analysis method (the Bunge-Roe method) [6].

3. EXPERIMENT

3.1 Methods of obtaining and preliminary preparation of samples

The subjects of investigations were alloys VT1-0 and VT6. The casting of VT1-0 alloy underwent hot rolling with symmetrical rollers. The resulting sheet of VT1-0 is shown in Figure 1. From the rolled sheet, samples were cut from center (sample 2) and edge (sample 1) of plate, with geometric dimensions of 40*40*0.5 cm. The geometry of the obtained samples and the sheet is shown in Figure 2. To conduct X-ray structural studies and perform laser shock processing aimed at removing near-surface layers, rolling defects, and oxides, mechanical processing of samples 1 and 2 was carried out.

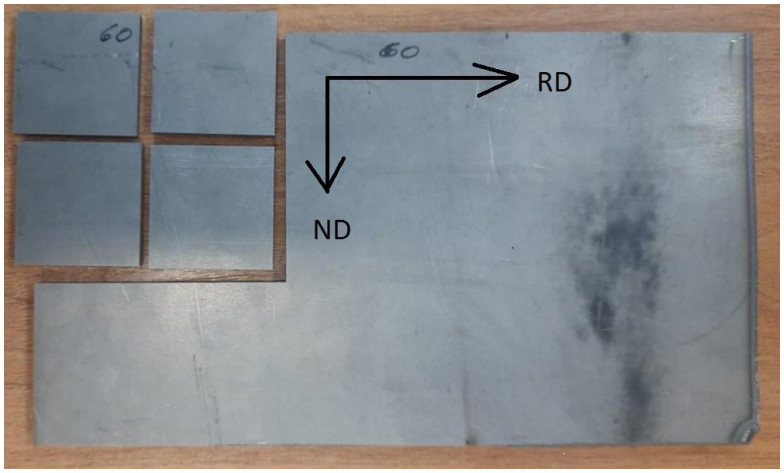


Fig. 2. Geometry of the sheet and the sample made of alloy VT1-0. (The figure shows the coordinate system of the sample relative to the sheet, where RD is the rolling direction, and ND is the normal direction.)

Samples of VT1-0 after polishing and laser shock processing are shown in Figures 3 and 4.

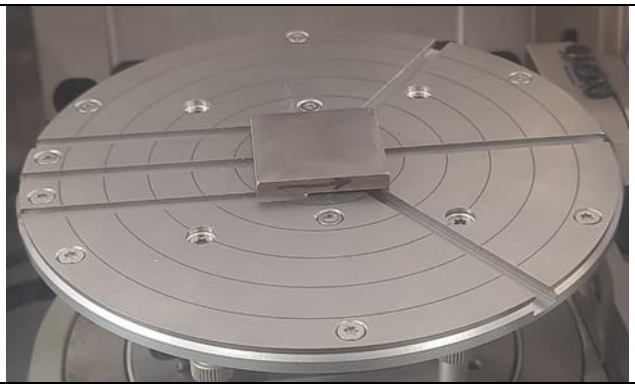


Fig. 3. Sample after polishing on the goniometer stage.

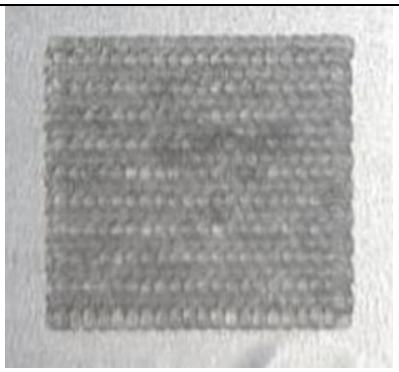


Fig.4. Sample with foil after laser shock processing.

Alloy VT6 was supplied in sheet form with geometric dimensions of 3 × 1000 × 1580 mm. From the obtained sheet, two plates were cut out from the center with the following geometric dimensions. The dimensions of obtaining samples were 40 × 40 × 0.5 mm.

3.2 Laser shock peening

In this study, samples made of alloys VT1-0 and VT6 were subjected to laser shock processing using solid-state Nd:YAG Beamtech SGR-Extra laser and robotic six-axis manipulator STEP SR50. The laser wavelength is 1064 nm, maximum pulse repetition frequency is 5 Hz, energy per pulse is 1 J and pulse duration is 10 ns. The robotic six-axis manipulator, with a payload capacity of 50 kg and a positioning accuracy of 0.25 mm, allows for automation of processing samples of arbitrary geometry.

During the processing, the manipulator automatically positions the workpiece in front of the laser beam so that the beam strikes the surface perpendicularly, after which it commands the laser machine to generate a pulse using transistor-transistor logic. The laser beam paths are generated using specialized software based on a three-dimensional model of the sample.

As a confining medium for the generation of shock waves, water was used. In this work, a square objective with a square spot with a side of 1 mm was used. The overlap density of spots was 0%. The processing was carried out at power density of 10 GW/cm². The processing diagram of the sample is shown in Figure 5.

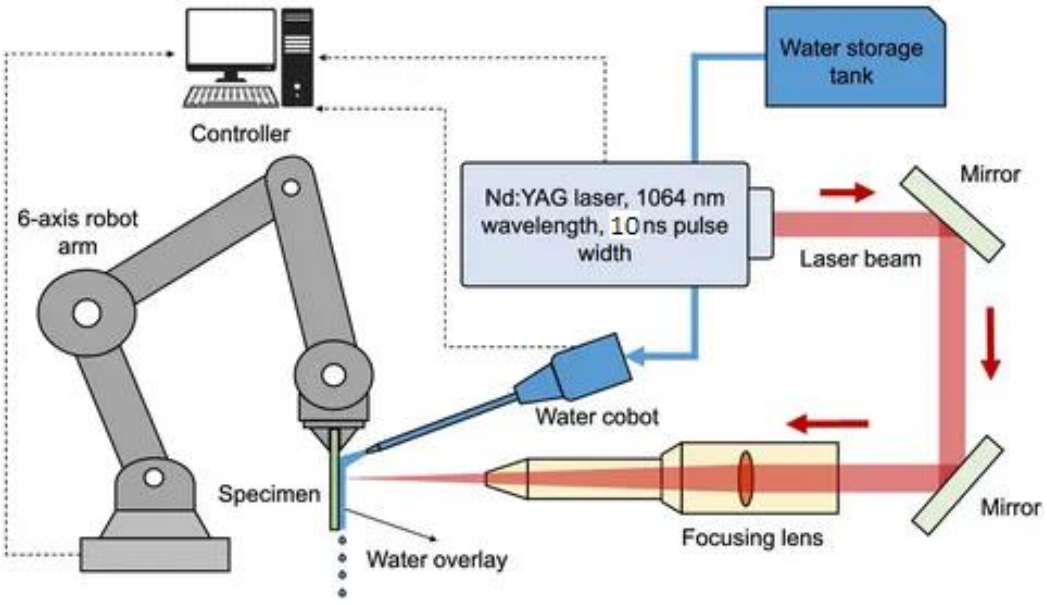


Fig.5. General scheme of sample processing during laser shock peening.

3.3 X-ray diffraction and texture measurements

The crystallographic texture of polycrystalline materials is investigated using methods such as neutron diffraction, electron microscopy or X-ray diffraction techniques. In this study, the latter method was employed.

Studies of titanium alloys VT6 and VT1-0 were conducted using multifunctional X-ray diffractometers Panalytical Empyrean and Rigaku Ultima IV (the diffractometers are shown in Figure 6) in Cu ($K_{\alpha 1}$ radiation) at U = 40 kV and I = 40 mA.

a

b

Fig.6. X-ray diffractometers Panalytical (a) and Rigaku Ultima IV (b).

The sample was positioned on the goniometric stage in the direction from the X-ray tube to the detector along the rolling plane. The sample was adjusted in height by scanning along the Z-axis so that it intercepted the center of the beam. A Ni filter was used to cut off the K_β radiation.

The research was conducted in 2 stages:

1. X-ray measurement

Slits with angles of 1/8º and 1/4º and 5 mm mask were used to obtain the X-ray spectrum. The spectrum was scanned over an angle range from 30° to 80°. By comparing the obtained spectra with the PDF-2 database, the phase composition of the resulting sample was established. Based on scanning results, positions of peaks were selected and refined for texture investigations.

The indices for texture investigations were selected according to the following rules:

1. plane with highest signal-to-background ratio
2. plane with small Miller indices.

2. Texture measurements

It is possible to measure only incomplete pole figures by XRD method. The incomplete pole figures were measured using the reflection method. Base of this method is that diffraction on the sample is fixed by a stationary detector due to rotation and tilting of the sample. To record the texture, measurements are made at a fixed Bragg angle 2Θ for the set of crystallographic planes (*hkl*). When transitioning to another system of planes, different angle is used, and the detector is moved to a different position. The reflection measurements were conducted within an azimuthal angle range from 0° to 360° with grid step of 5°, and radial angle Φ ranging from 0° to 70° with grid step of 5°. The measurement time for one position was 18 seconds. For texture measurements, a slit system with angles of 0.25° and 0.5° and mask of 10 mm was used.

The registration of incomplete pole figures was carried out by recording the intensity of the reflected rays for a given position of the sample (in coordinate system). The measurement process was repeated until the entire range of angles was measured.

In the X-ray reflection method, pole figures can be recorded up to Φ = 70°. This phenomenon is related to the defocusing effect. The main reason for this effect is decrease in area of studied spot as angle Φ increases. As a result, the intensity of the beam decreases at larger angles. To take into account the defocusing effect, the corrective factors are introduced. In this work, defocusing is accomplished by measuring a reference sample.

3.4 PF calculations in the MTEX software package

MTEX is an open-source Matlab toolkit for analyzing and modeling crystallographic textures using EBSD or X-ray data [7]. In this work, it was used to calculate the complete PF. The calculations are performed by the harmonic method.

To run the calculations, the following information must be set:

1. A space group of a crystal lattice and its parameters (in this work α-*Ti* have the space group 622 and unit cell parameters $a = b = 2.951A$, $c = 4.686\ A$).
2. Symmetry of the samples
3. Sample coordinate system
4. Indices of crystallographic planes for which measurements were made.
5. Incomplete pole figures (uploaded as ASCII files).
6. Data of incomplete pole figures for the reference sample for defocusing analysis.

The calculation procedures include the following stages:

1. Preparation and loading of experimental incomplete pole figure (InPF) data.
2. Conducting experimental (or theoretical defocusing) using the reference sample.
3. Normalization NPF
4. Calculation ODF
5. Calculation PF from ODF
6. Comparison of recalculated pole figures and NPF using the RP factor.

In order to account for the deviations of recalculated pole figures from experimental ones, the RP factor is introduced. This parameter allows the evaluation of the error in the recalculated pole figures. The analytical expression for RF calculating is given by the following equation:

$$RP = \sqrt{\frac{\sum_{i=1}^{m}(I_{meas,i} - I_{calc,i})^2}{\sum_{i=1}^{m} I_{meas,i}{}^2}}$$

4. RESULT

The X-ray diffraction pattern of the VT1-0 sample and its analysis showed that the surface layers of material consist of phase α-Ti (Fig.7). The crystalline lattice of alpha-titanium has a space group of symmetry 622. This is a unit cell with parameters a = 2.951 A, c = 4.686 A (from crystallographic database). The angles between basis vectors of the unit cell α = 90°, β = 90° and γ = 120°. The X-ray measurement method does not allow for the determination of phases with a concentration of less than 5%.

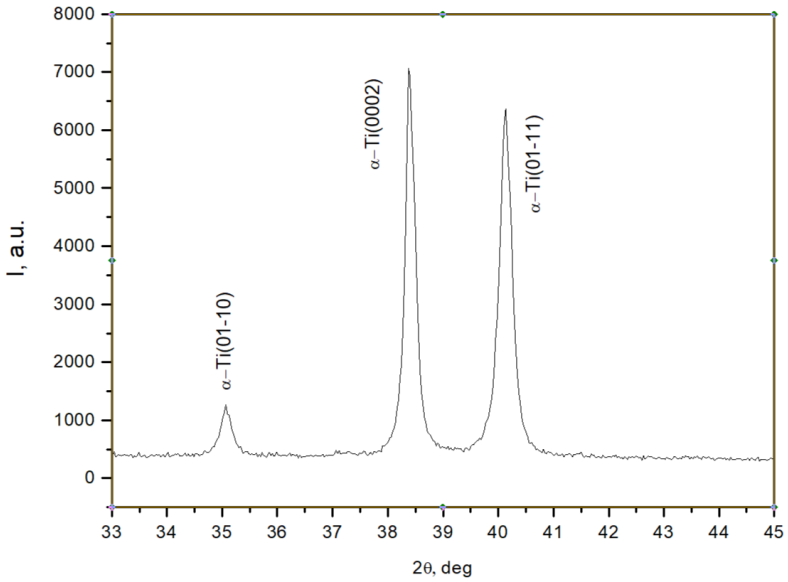


Fig.7. X-ray diffraction pattern of the sample α-Ti.

Crystallographic planes (002), (010) and (011) were used to measure pole figures. These planes make the interpretation of the crystallographic texture most accurate. It took approximately 5 hours and 30 minutes to measure pole figure for each crystallographic plane.

The recalculated pole figures using the MTEX program for the VT1-0 alloy before and after LSP are presented in Figures 8 and 9. The calculations of the pole figures took into account the correction for experimental defocusing using a reference sample. The error value (determined by the parameter RP) considering defocusing does not exceed 5.2% for all pole figures (Table 1).

Qualitatively, the patterns of all complete pole figures coincide. The figures 8 and 9 show, that maximum pole density is located at the center (for PF (002)). So, crystals of α-Ti with a small degree of texture scattering are spaced in the plane to the rolling direction of the sheet.

The quantitative characteristic of texture sharpness is represented by the parameter mrd (units of isotropic distribution). Calculations have shown that the crystallographic texture of α-Ti is relatively weak, with a value of 2.6 mrd.

The texture of the sample in center, on edge and in the intermediate position different quantitatively (Fig.8-10). The values of pole density for samples at the center, in an intermediate position, and at edge of the sheet are 2.6, 2.9, and 3.2 mrd, respectively. So, in the sample the crystallographic texture is heterogeneous.

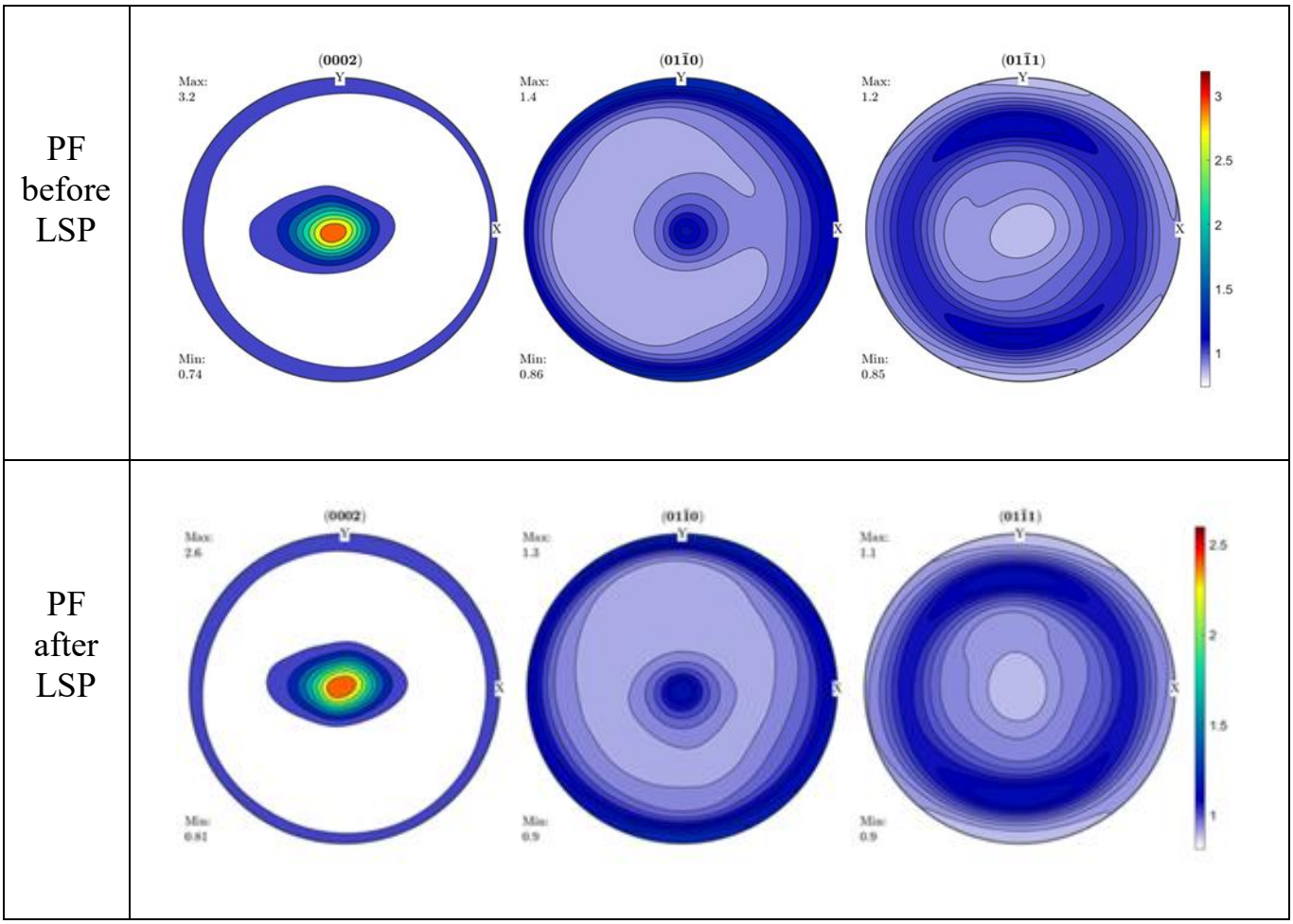


Fig.8. PF of the sample from edge of the sheet before and after LSP(made in the MTEX program with taking into account the experimental defocusing)

A comparison of the PF (Fig.8-9) shows that LSP leads to a decrease in texture sharpness in case when there is an impact on the edge of the sheet (mrd decreases from 3.2 to 2.6). In the case when laser is applied to the center of the sheet, the texture does not change much (mrd varies from 2.6 to 2.4).

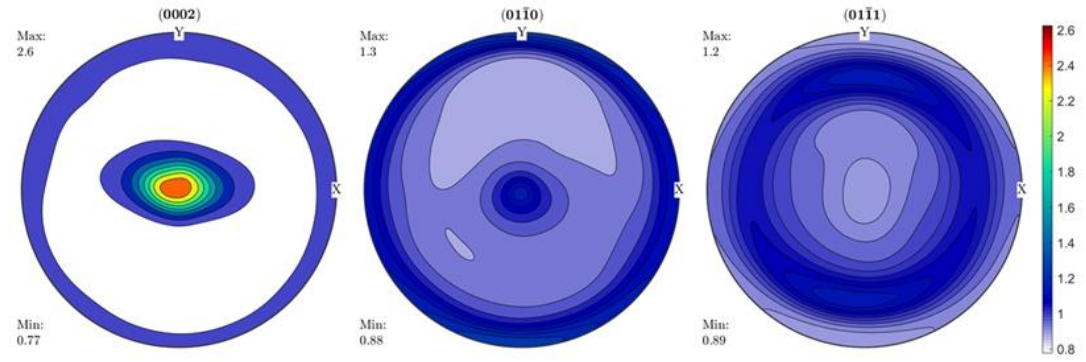
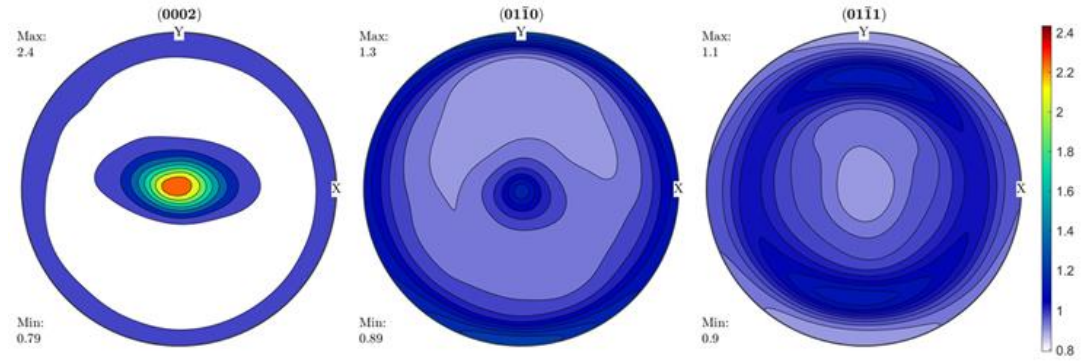
PF before LSP	
PF after LSP	

Fig.9. PF of the sample from center of the sheet before and after LSP (made in the MTEX program with taking into account the experimental defocusing)

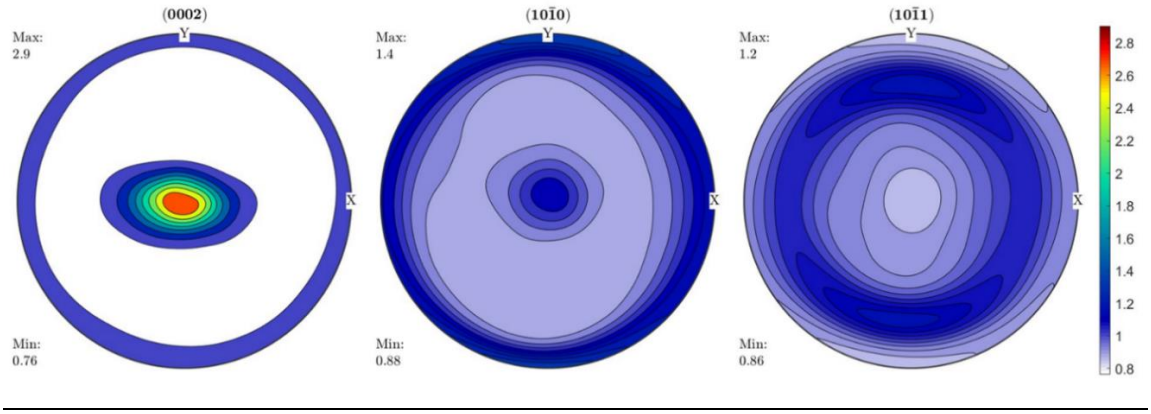
PF			
RP	4.27 %	3.21%	---

Рис.10. PF of α-Ti in the intermediate position between the edge and center of the sample.

Based on results of the 2θ scanning conducted in range from 20° to 70°, it was found that surface layer of the VT6 sample contains only the α-Ti phase.

The crystallographic planes (0002), (10-10), and (10-11) were selected for measurement. These planes were chosen based on their highest reflection intensity.

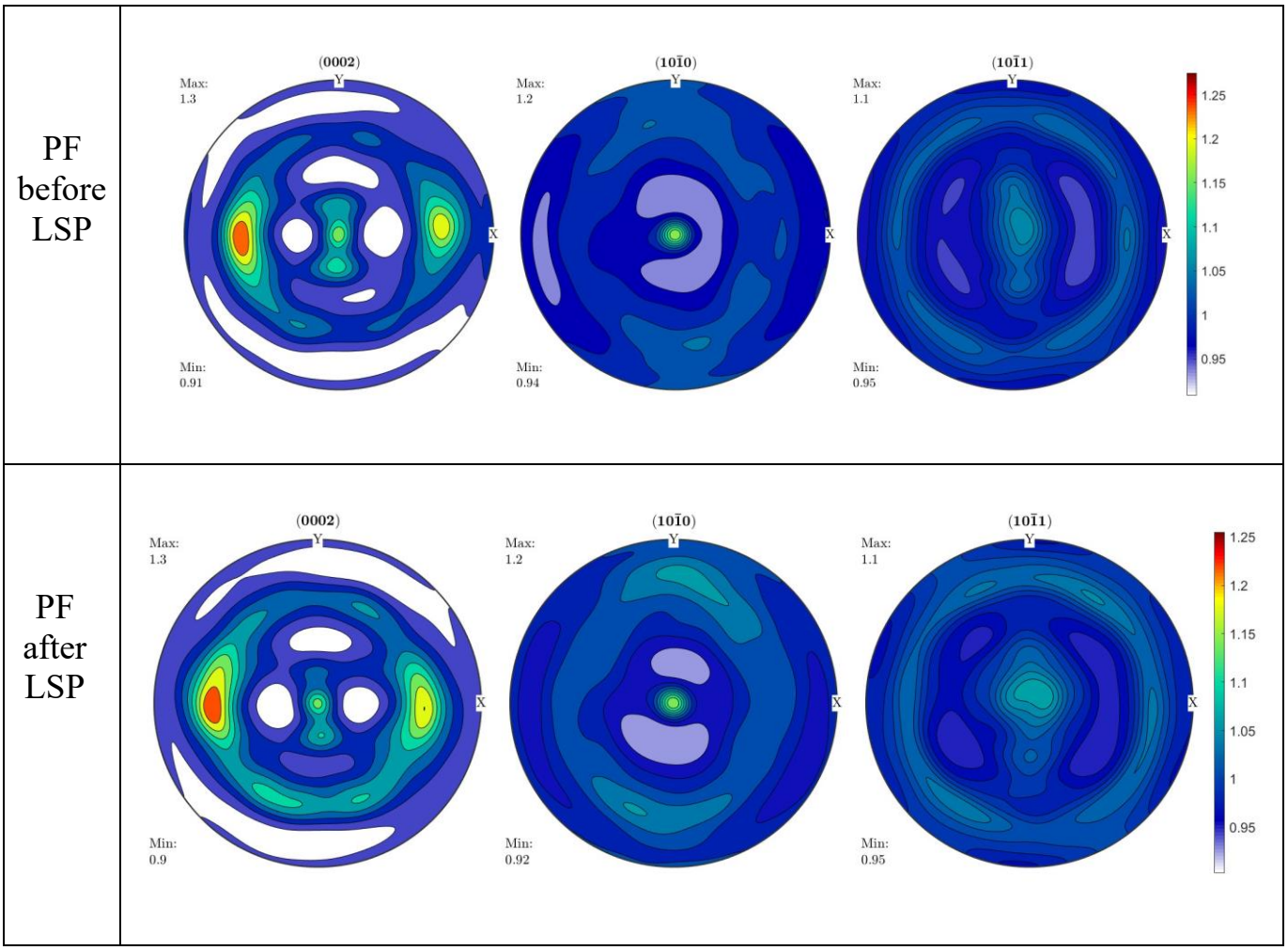


Fig.11. PF of α-Ti in the center of rolled sheet (VT6) alloy before and after LSP.

The results of calculation for α-Ti in the VT6 alloy based on the 3 pole figures are shown in figure 11. The PFs of α-Ti before and after Laser Shock Processing (LSP) for the VT6 qualitatively and quantitatively (within the calculation errors) have no changes. It is evident that the pole density maximum for the base plane is 1.3 mrd. All maximum and minimum values of the pole densities are close to 1 mrd, indicating the absence of grains with a preferred orientation.

5. APPENDICES

We calculated the pole figures measured experimentally for the reference (powder) sample to take into account the defocusing effect. The calculation results for 3 and 2 NPF without correcting of the obtained data are shown in Figures 12 and 13.

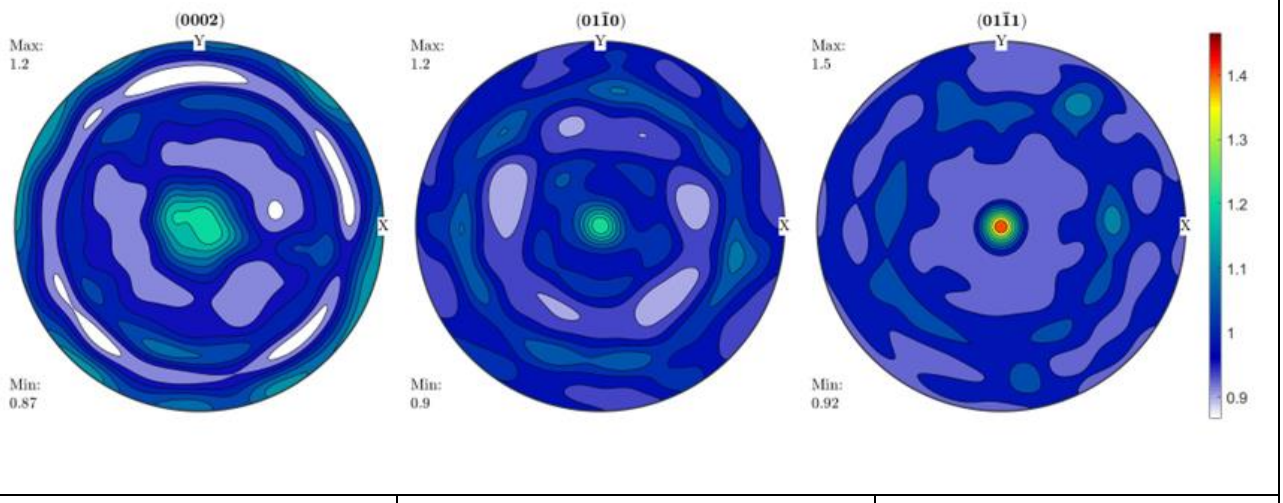
PF			
RP	9.4 %	13%	15 %

Fig.12. The recalculated pole figures for the reference sample based on 3 PF.

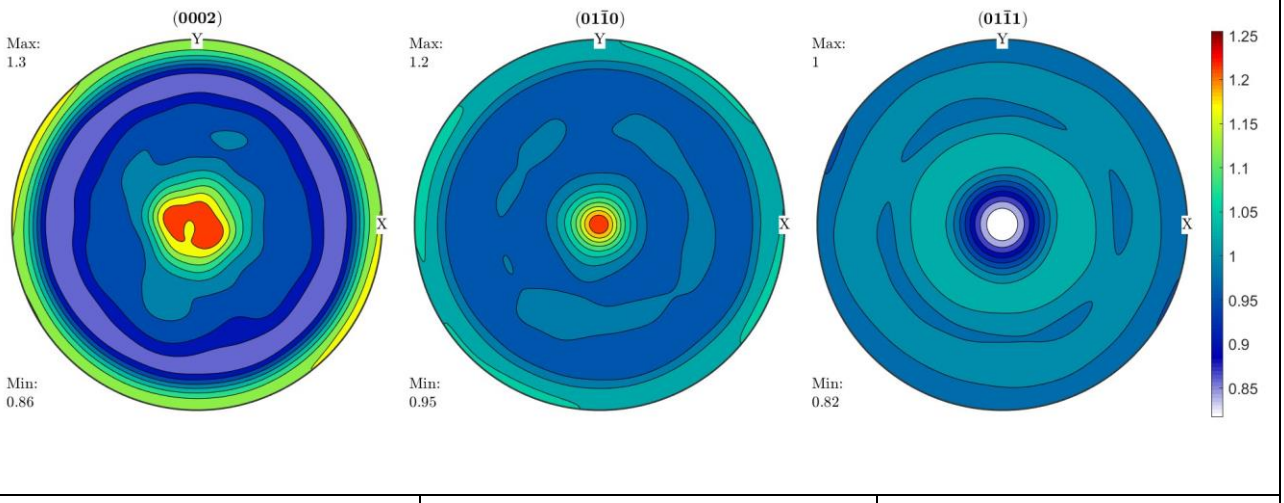
PF			
RP	6.5 %	10.4 %	---

Fig.13. The recalculated pole figures for the reference sample based on 2 PF.

The pole figures of the reference sample (Figures 3 and 4) show that there are grains with well-defined orientations in the powder. The intensity values corresponding to these grains were removed from the data. It is known that, in the reference material, the intensity values do not depend on the rotation angle of the sample around its normal (after removing the irrelevant values, all quantities were averaged at the same angle). The resulting recalculated pole figures are shown in Figure 14.

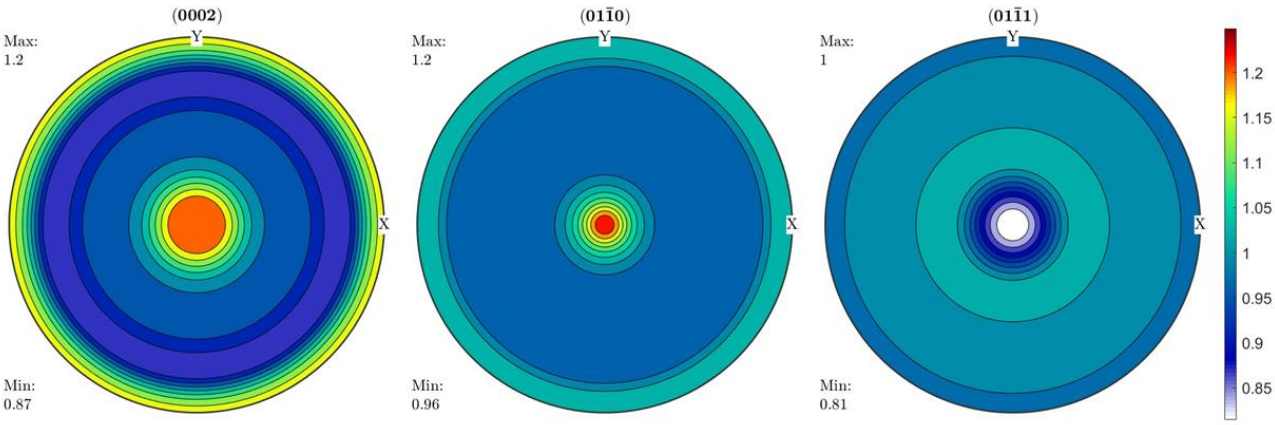
PF			
RP	6 %	10 %	---

Fig.14. Recalculated pole figures for the reference sample with taking account outliers

6. CONCLUSION

X-ray phase analysis showed that samples of alloys VT1-0 and VT6 after hot rolling consist of hexagonal phase α-Ti. Other phases are present in small concentrations.

The crystals of` phase α-Ti in VT1-0 are arranged parallel to the rolling plane, which is typical for rolled materials. Studies have also shown that textures sharpness increases from the center to edge of sample. The pole density values for VT1-0 from center to edge of the sheet increased from 2.6 to 3.2 mrd, respectively. The texture heterogeneity across the sample is most likely due to the fact that the rolled sheet has small geometrical size. There were no qualitative changes in the pole figures.

In VT-6 alloy, maximum pole density is 1.3mrd for (0002). So, in the VT-6 alloy, crystallographic texture is very weak. After LSP can see that crystallographic texture does not change.

The XRD results showed that there is no change of crystallographic texture in VT1-0 and VT6 after LSP. Nevertheless, EBSD analysis is necessary to understand the mechanism of LSP impact accurately.

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