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Recrystallization temperature influence upon texture evolution of a SPD processed Ti-Nb-Ta-Zr-O alloy

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Abstract. The present study investigates the texture features occurred during recrystallization of a Ti-29Nb-9Ta-10Zr-0.2O (wt.%) alloy processed by multi-pass cold-rolling, up to 90% thickness reduction. Data concerning alloy component phases and the lattice parameters of identified phases were obtained and analysed for all thermo-mechanical processing stages. Crystallographic texture changes occurred during alloy thermo-mechanical processing (cold-rolling and recrystallization), were investigated using X-ray diffraction; by acquiring the pole figures data of the main β-Ti phase. Data concerning observed texture components and texture fibers was analysed using φ1 - Φ - φ2 Bunge system in φ2 = 0° and 45° sections. The γ textural fiber was analysed for all thermo-mechanical processing stages.

1. Introduction

Titanium alloys are extensively used in a variety of applications due to their good mechanical properties and corrosion resistance [1]. Comparing with α and α+β titanium alloys, the β Titanium alloys possess excellent properties such as higher strength and better plasticity [2]. In the last years, β-type Titanium alloys, containing β-isomorphous non-toxic alloying elements, have received much attention due to their good mechanical properties as well as high corrosion resistance and biocompatibility. The addition of β-isomorphous non-toxic alloying elements, such as niobium (Nb), tantalum (Ta) and zirconium (Zr), acts as β-phase stabilizers and compounds formation suppressors, improving alloy's mechanical properties [3-5].

Complementary to chemical composition, the thermo-mechanical processing is often applied in order to obtain a desired combination of mechanical properties based on obtaining various microstructures, containing different component phases in different volume fractions.

In the case of β-type Titanium alloys used in medical applications, especially in osseous implantology, a low elastic modulus is desired. In order to decrease the elastic modulus, microstructures consisting in a mixture of both α"-Ti and β-Ti phases must be obtained, due to the lower elastic modulus of the α″-Ti phase in comparison with parent β-Ti phase [6-7].

Another way to stir the mechanical properties consists in obtaining of a textured microstructure, due to the strong influenced of crystallographic anisotropy upon mechanical properties. In the recent years large efforts were dedicated to controlling the developed crystallographic texture by an adequate applied thermo-mechanical processing route.

The aim of the present paper is to present obtained results in respect to developed crystallographic texture in the case of a Ti-29Nb-9Ta-10Zr-0.2O (wt%) alloys. Structural changes occurred during thermo-mechanical processing were investigated using X-ray diffraction.

2. Methods

As synthesis technique for the Ti29Nb-9Ta-10Zr-0.2O (wt%) alloy the melting in cold crucible induction in levitation was used. A FiveCeles MP25 synthesis equipment was used in order to obtain the Ti29Nb-9Ta-10Zr-0.2O (wt%) alloy. Due to the large difference in melting temperatures of alloying elements (Ti: 1660°C; Nb: 2468°C; Ta: 2996°C; Zr: 1855°C) two re-melts were performed in order to assure the alloy's chemical homogeneity.

The Ti-29Nb-9Ta-10Zr-0.2O alloy thermo-mechanical processing route consisted in the following stages: After alloy synthesis, in order to increase the alloy homogeneity, a homogenisation treatment (in high vacuum) was performed at 950°C for 6 hours. In order to obtain a refined microstructure a first cold-rolling was applied using a total thickness reduction of 60%, followed by a recrystallization treatment applied to remove the strain-hardening effects developed during cold deformation. The recrystallization treatment (in high vacuum) was performed at 780°C for 30 min. Recrystallized samples were water quenched in order to obtain a bi-modal microstructure consisting of parent β-Ti phase and temperature-induced α"-Ti phase.

A second cold-rolling, with a thickness reduction of 90%, was performed in order to obtain the first investigated structural state (structural state 1). The total applied equivalent plastic strain was close to $\varepsilon = 2.38$, above the threshold of $\varepsilon = 2$, which represents the limit for a *severe plastic deformation* (SPD) processing.

The thermo-mechanical processing was continued with a second recrystallization treatment (in high vacuum) performed at 780°C (structural state 2) and at 880°C (structural state 3), for 30 min and water cooling.

All obtained structural states, 90% cold-rolled, cold-rolled and recrystallized at 780°C and cold-rolled and recrystallized at 880°C were subjected to XRD characterization.

The XRD characterization was performed using a Philips PW 3710 diffractometer, with Cu k-alpha ($\lambda = 1.5406$ Å), in order to determine the alloy constituent phases, phase characteristics and also the pole figures. The (110), (200) and (211) Pole Figures (PF's) of the β-Ti phase were measured. The PF's were collected in 5° increments: 0°-χ-85°, 0°-ϕ-355°. The texture analysis was performed using MTEX v3.5.0 software package. The PF's raw data was analysed using Gaussian distribution and Ghost correction. Inverse Pole Figures (IPF's) and Orientation Distribution Functions (ODF's) were calculated.

Taking into consideration the low intensity of α"-Ti phase peaks in comparison with β-Ti phase peaks and the α"-Ti phase peaks positions, makes the recording of α"-Ti phase PF's very difficult, for this reason only the texture of β-Ti phase is analysed and presented here.

3. Results and discussion

3.1. XRD measurements

In order to compute the recorded XRD spectra, the β-Ti phase was indexed in BCC system - *Im-3m*, while the α"-Ti phase was indexed in orthorhombic system – *Cmcm*. In the XRD spectra fitting procedure a pseudo-Voight diffraction line profile was used due to the introduced effects of cold-rolling processing in materials microstructure. The recorded XRD spectra of 90% cold-rolled state, presented in figure 1-a, shows the presence of (110), (200) and (211) diffraction lines corresponding to β-Ti phase and the (020), (002), (111), (022), (200), (130), (131), (113) (220), (202) and (023) diffraction lines corresponding to α"-Ti phase.

Figure 1-b shows a detailed zoom corresponding to $2\theta = (67° - 75°)$, can be observed that the convolution of α"-Ti (131), α"-Ti (113), β-Ti (211), α"-Ti (220), α"-Ti (202) and α"-Ti (023) diffraction lines accurately describe the recorded XRD spectra. Can be observed that the convolution of all β-Ti and α"-Ti diffraction lines accurately describe the recorded XRD spectra.

Based on diffraction lines positions (2θ) and assumed phases crystalline systems, the lattice parameters corresponding to β-Ti and α”-Ti phases were computed, with a precision of ± 0.01. It was obtained that in the case of β-Ti phase the lattice parameter was: a = 3.29Å, while in the case of α”-Ti phase the lattice parameters were as follows: a = 3.21Å, b = 4.71Å and c = 4.61Å.

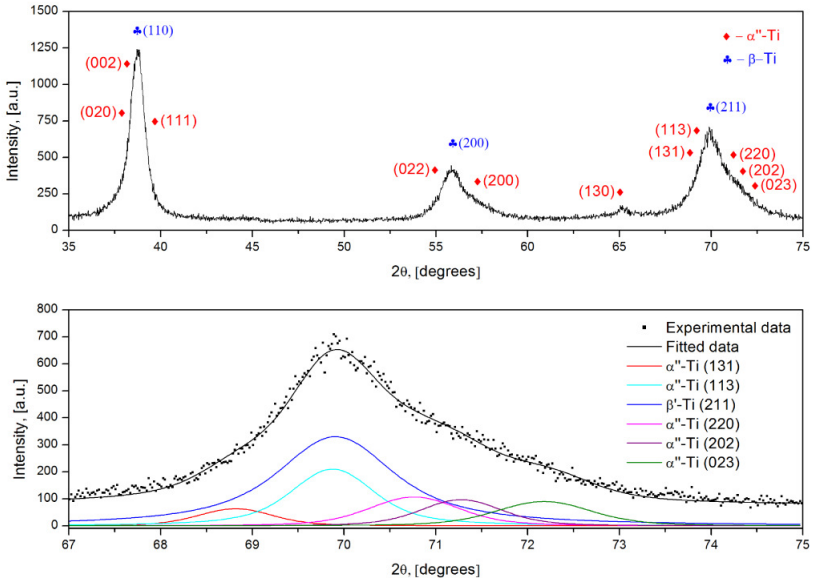

Figure 1. XRD recorded spectra in the case of 90% cold-rolled Ti-29Nb-9Ta-10Zr-0.2O (wt%) alloy (a); detailed XRD spectra corresponding to 2θ = (67° - 75°) (b).

The recorded XRD spectra corresponding to recrystallization at 780°C (structural state 2), presented in figure 2-a, shows the presence of same β-Ti and α”-Ti phases. In the XRD spectra fitting procedure a Lorentz diffraction line profile was used due to the introduced effects of recrystallization in materials microstructure.
Figure 2-b shows a detailed zoom corresponding to 2θ = (67° - 72°) were can be observed that the convolution of α”-Ti (131), α”-Ti (220), β-Ti (211), α”-Ti (202) and α”-Ti (113) diffraction lines accurately describe the recorded XRD spectra.
Similar with the previous case, based on diffraction lines positions (2θ) and assumed crystalline systems, the lattice parameters corresponding to β-Ti and α”-Ti phases were computed. It was obtained that in the case of β-Ti phase the lattice parameter was: a = 3.31Å, while in the case of α”-Ti phase the lattice parameters were as follows: a = 3.31Å, b = 4.76Å and c = 4.66Å.
Same observed diffraction lines in respect to both β-Ti and α”-Ti phases were detected also in the case of recrystallization performed at 880°C (structural state 3) (figure 3-a). The detailed zoom corresponding to 2θ = (69° - 71°) (figure 3-b) shows that the constituent diffraction lines accurately describe the recorded XRD spectra. The computed lattice parameters corresponding to β-Ti phase showed a lattice parameter: a = 3.285Å, while in the case of α”-Ti phase the computed lattice parameters were as follows: a = 3.28Å, b = 4.73Å and c = 4.63Å.
Comparing the lattice parameters of α”-Ti phase obtained in the case of mechanical (cold-rolling) processing with the ones obtained in the case of thermal (recrystallization) processing, a large difference can be detected in respect to lattice parameter *a* (a = 3.21 Å – mechanical processing; a = (3.31 - 3.28) Å – thermal processing), this large difference corroborate with the specifics of processing method, suggests that two formation mechanisms are involved in α”-Ti phase formation. In the case of mechanical processing the

α”-Ti phase is formed due to a stress induced transformation while in case of thermal processing the α”-Ti phase is formed due to a thermal induced transformation.

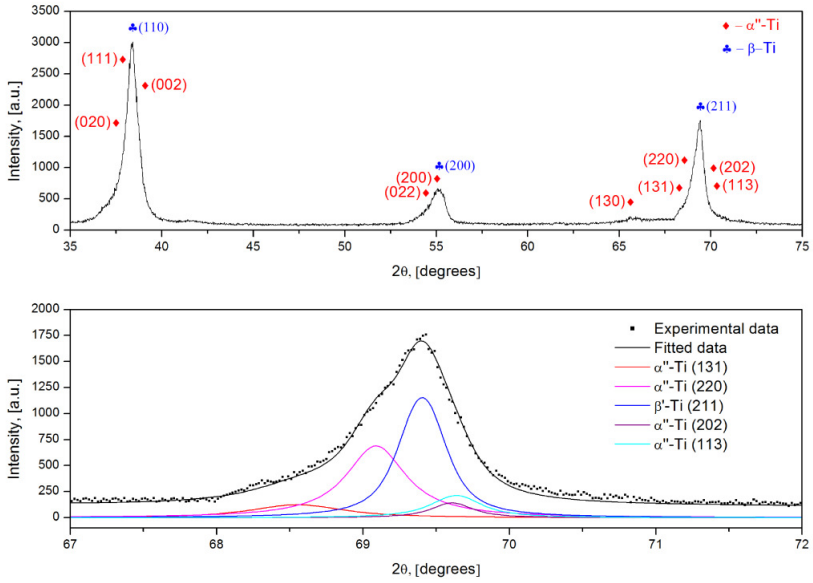

Figure 2. XRD recorded spectra in the case of recrystallized at 780°C (a); detailed XRD spectra corresponding to 2θ = (67° - 72°) (b).

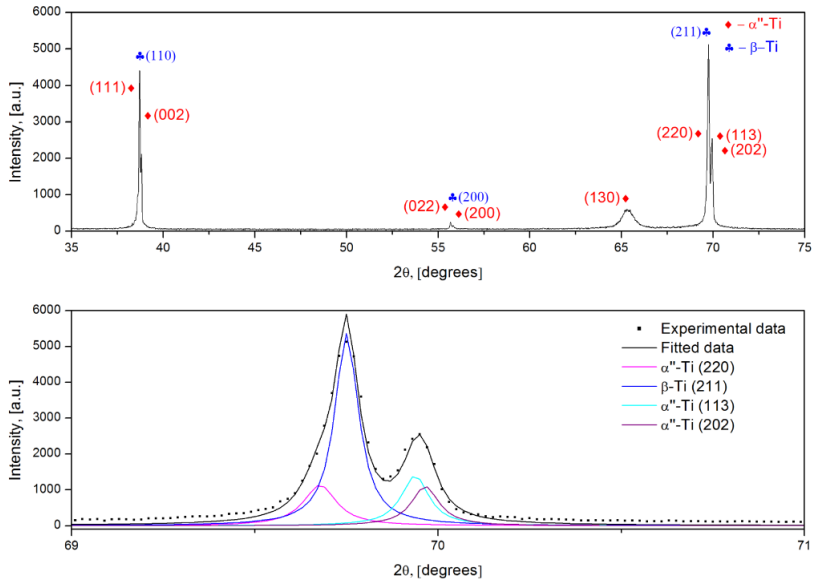

Figure 3. XRD recorded spectra in the case of recrystallized at 880°C (a); detailed XRD spectra corresponding to 2θ = (67° - 72°) (b).

3.2. Texture measurements

In order to calculate the IPF’s and ODFs, few assumptions were made, first, sample crystalline symmetry was indexed in cubic *Im-3m* system, and second, sample geometric

symmetry indexed in orthorhombic *mmm* system. The IPF's and ODF's were computed using experimental recprded (110), (200) and (211) pole figures of the β-Ti phase and processed using *de la Vallée Poussin* kernel function, Gaussian distribution, Ghost correction and intensities normalization. During IPF's and ODF's computation the misorientation was set to 3°.

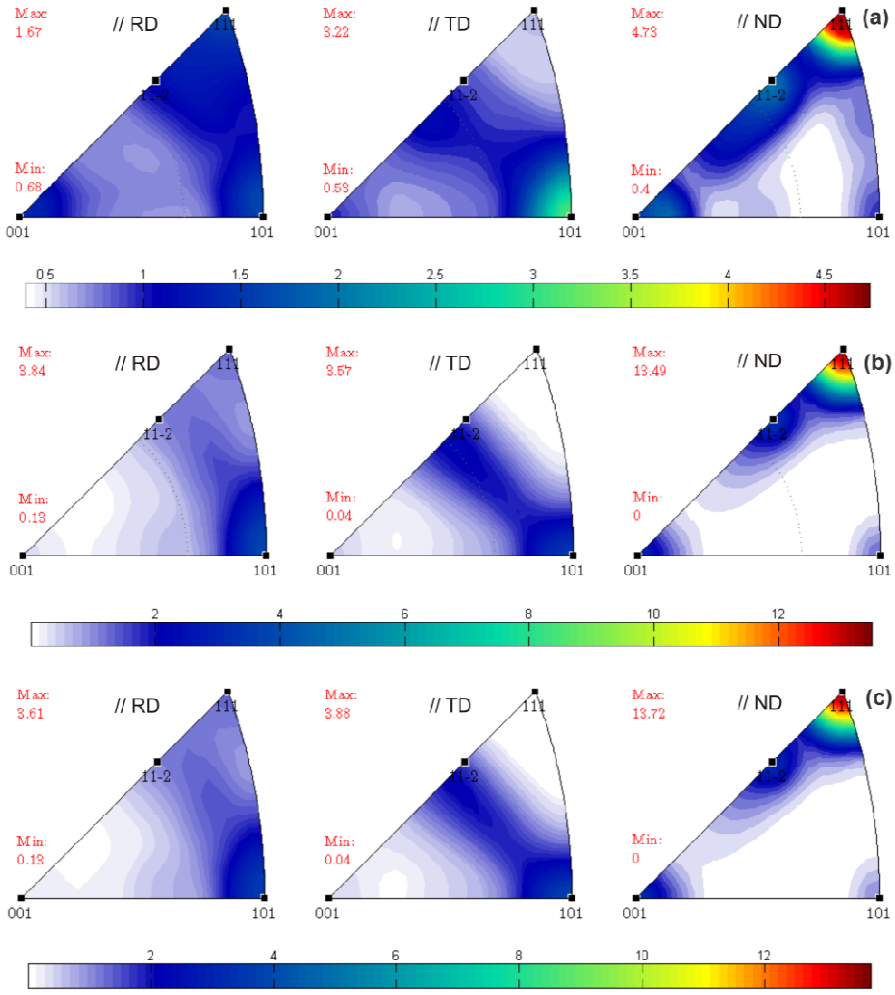

Figure 4. Representation of Inverse Pole Figures (IPF's); a – 90% cold-rolled state; b – 780°C recrystallized state; c – 880°C recrystallized state.

The analyse of developed crystallographic texture by terms of Inverse Pole Figure (IPF) is presented in figure 4, corresponding to 90% cold-rolling (figure 4-a), recrystallization at 780°C (figure 4-b) and recrystallization at 880°C (figure 4-c). The IPF's shows how a selected direction in the sample reference system is distributed in the crystal reference system.
Analysing the IPF's one can observe that in the case of 90% cold-rolled state (figure 4-a) [111] crystallographic direction is most likely parallel to sample normal direction (ND), the [111]//ND pair reaching an intensity close to 4.7 MRD (MRD – Multiple of a Random Density). In the case of RD and TD directions most intense observed pairs are [101]//RD and [101]//TD, with an intensity of 1.6 MRD and respectively 3.2 MRD. In the case of recrystallized states can observe that the [111]//ND pair is reaching the highest intensity, close to 11.2 MRD in the case of recrystallization at 780°C (figure 4.b) and 17.1 MRD in the case of recrystallization at 880°C (figure 4-c). Other formed crystallographic pairs are:

[101]//RD, [101]//TD with transition to [112]//TD and [112]//ND. All other formed crystallographic pairs show negligible intensities in comparison with [111]//ND pair.
If comparing the intensity of [111]//ND pair observed in the case of 90% cold-rolled state, situated close to 4.7 MRD, with the one observed in the case of recrystallization at 780°C state, situated closed to 13.4, can be observed that the increase is approximately three times bigger, suggesting that a stronger texture is obtained after thermal processing in comparison with mechanical processing.
Figures 5, 6 and 7 show the representation of computed ODF's in the case of 90% cold-rolled state (figure 5), recrystallization at 780°C (figure 6) and recrystallization at 880°C (figure 7). As observed, in all cases the highest orientation densities are obtained in the case of $\varphi_2 = 45°$ section.

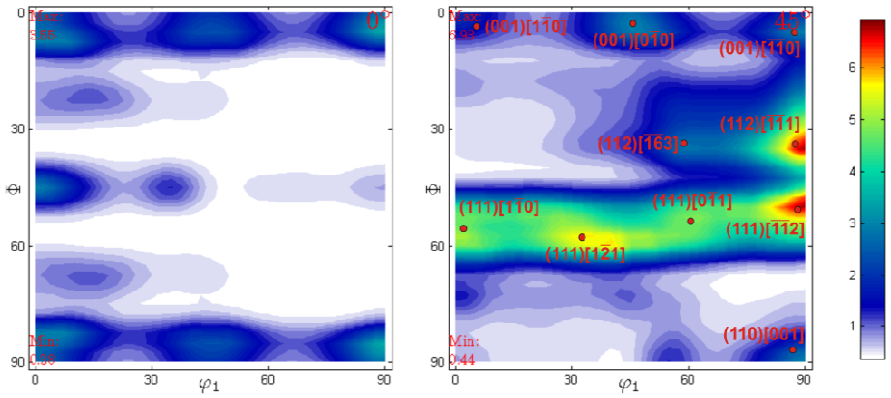

Figure 5. Representation of Orientation Distribution Functions (ODF's) corresponding to 90% cold-rolled state; a – ODF section: φ2 = 0°; b – ODF section: φ2 = 45°.

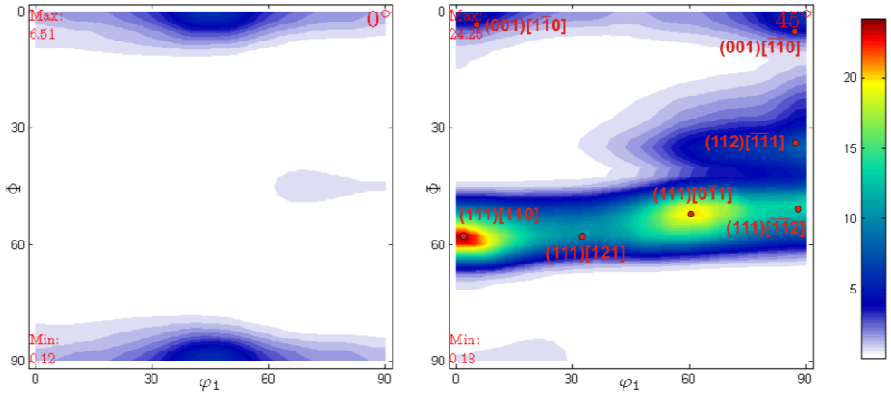

Figure 6. Representation of Orientation Distribution Functions (ODF's) corresponding to recrystallization at 780°C state; a – ODF section: φ2 = 0°; b – ODF section: φ2 = 45°.

In the case of 90% cold-rolled state (figure 5) can be observed that the maximum orientation density is obtained in the case of texture components $(112)[\bar{1}\bar{1}1]$ and $(111)[\bar{1}\bar{1}2]$ with an orientation density close to 6.9 MRD. Other observed texture components were as follows: $(111)[1\bar{1}0]$, $(111)[1\bar{2}1]$, $(111)[0\bar{1}1]$ and $(111)[\bar{1}\bar{1}2]$, all belonging to the γ-fibre.
In the case of recrystallized at 780°C and 880°C states, one can observe that the maximum orientation density is obtained in the case of γ-fibre components $(111)[1\bar{1}0]$ and $(111)[0\bar{1}1]$.

In both cases the highest orientation density is recorded in the case of $(111)[1\bar{1}0]$ texture component, close to 24.2 MRD in the case of 780°C (figure 6) and close to 25.9 MRD in the case of 880°C (figure 7).

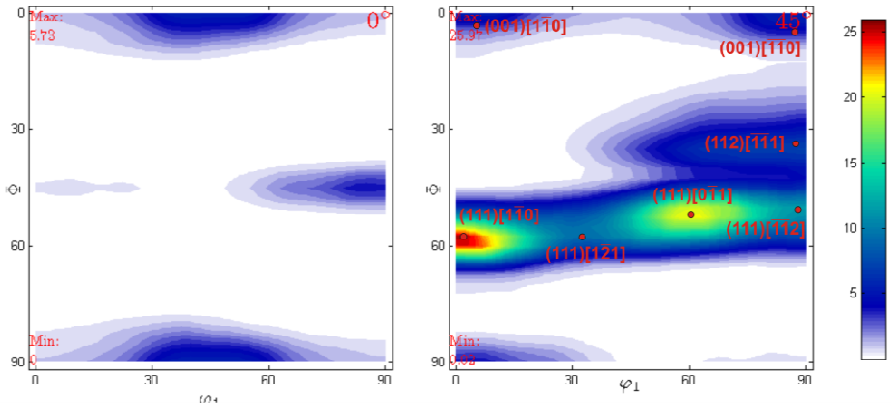

Figure 7. Representation of Orientation Distribution Functions (ODF's) corresponding to recrystallization at 880°C state; a – ODF section: φ2 = 0°; b – ODF section: φ2 = 45°.

If the orientation density of the γ textural fibre is plotted (figure 8), can be observed that the mechanical processing favours the $(111)[1\bar{2}1]$ and $(111)[\bar{1}\bar{1}2]$ texture components, both showing an orientation density between (5.5 – 6.9) MRD. The result of thermal processing in terms of developed texture components shows that the thermal processing favours the texture components $(111)[1\bar{1}0]$ and $(111)[0\bar{1}1]$, reaching almost equals orientation densities in the case of both recrystallization temperatures, with an orientation density between (18 – 26) MRD, proving, on one hand, that by thermal processing the obtained texture is much more stronger in comparison with mechanical processing, and by other hand, that the recrystallization performed at temperatures between 780°C and 880°C show almost no differences upon developed texture components.

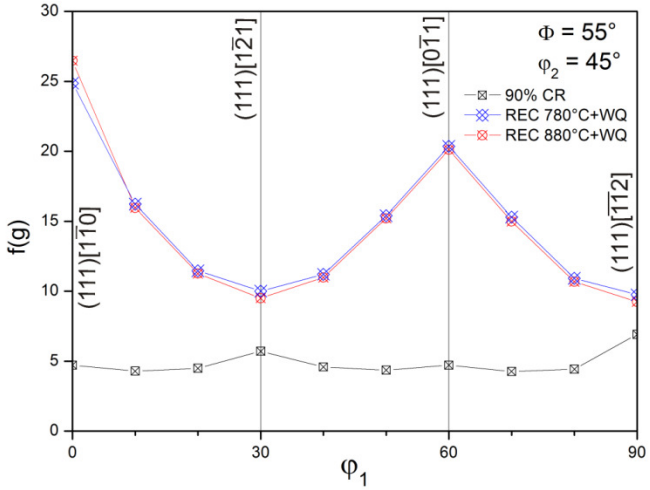

Figure 8. γ-fiber developed during thermo-mechanical processing

4. Conclusions

The present study investigates the developed microstructural features, in terms of constituent phases and crystallographic texture, in the case of thermo-mechanical processed Ti-29Nb-9Ta-10Zr-0.2O (wt.%) alloy. The conclusions of this study can be are summarized as follows:

- the XRD investigations revealed that the phase structure of all studied structural states consist of a mixture of β-Ti and α″-Ti phases. The α″-Ti phase being obtained by *stress-induced transformation* in the case of 90% cold-rolled state and by *temperature-induced transformation* in the case of recrystallized states;
- in the case of 90% cold-rolled state the most intense crystallographic texture components are $(112)[\bar{1}\,\bar{1}1]$ and $(111)[\bar{1}\,\bar{1}2]$, while in the case of recrystallized states the most intense crystallographic texture components are $(111)[1\,\bar{1}0]$ and $(111)[0\,\bar{1}1]$, both belongings to γ textural fibre;
- the maximum orientation density (texture strength) in the case of recrystallized states is fourth times more intense than in the case of cold-rolled state.

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