

# Superelastic alloy Ti-22%Nb-6%Zr: X-ray study of deformation features

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**Abstract.** In this paper features of deformation of superelastic Ti-22%Nb-6%Zr alloy were studied. Using X-ray method of generalized pole figures for study orientation dependence of martensite transformation in superelastic Ti-22%Nb-6%Zr alloy it was shown that: under compression the strain hardening due  $\beta \rightarrow \alpha''$  transformation is significantly greater in grains with rolling plane  $\{100\}$  than in grains with a rolling plane  $\{111\}$ ; the lattice microdeformation at  $\beta$ - and  $\alpha''$ - phase grains under loading is redistributed so that it extremely assist the sample macrodeformation (most of all); most intense  $\beta \rightarrow \alpha''$  transformation occurs in grains, where  $\Delta d / d_{av} > 0$ , most prone to increasing of its lattice volume. Diagrams of the cross-correlation substructural parameters built on the basis of generalized pole figure indicate that in loaded sample the deviation of interplanar space from the weight average level promotes the activation of martensite transformation at which the X-ray line broadening is minimal.

## 1. Introduction

Superelastic alloys are now popular objects of studies both because of interesting scientific aspects and owing to perspective practical applications. The mechanism of superelasticity consists in continuation of the elastic deformation by  $\sim 0.2\%$ , connected with interaction of neighboring atoms, by reversible elastic deformation by  $2\div 3\%$  and more due to martensitic transformations (MT). The given paper is devoted to X-ray study of the superelastic alloy Ti-22%Nb-6%Zr (at. %) (TNZ) using methods of texture analysis. Several detailed papers on this alloy were published recently in different countries [1-4]. In particular, it was shown that superelasticity of TNZ alloy is conditioned by  $\beta \leftrightarrow \alpha''$  MT, where  $\beta$ -phase has BCC crystalline lattice and  $\alpha''$ -phase – orthorhombic lattice. It was found as well that  $\beta \rightarrow \alpha''$  MT realizes with an increase of the elementary cell volume [4]. But the approach of authors was directed mostly to integral description of material and they did not split it into separate texture components, playing different roles in behavior of the alloy by MT. Since TNZ alloy is used usually in the rolled state, its samples have always the developed crystallographic texture and we need to take into account significant regular substructure inhomogeneity, accompanying formation of this texture. In the course of preliminary experiments it was shown that in TNZ alloy MTs and connected with them superelasticity take place at the room temperature.

The foil of superplastic alloy Ti-22%Nb-6%Zr (at. %) was rolled down to thickness of  $\sim 0.2$  mm. Then the foil was deformed in different manners in order to observe reversible MTs: (1) the foil was located in the special set for the X-ray diffractometer, where it could be stretched under X-ray irradiation; (2) the foil was bent by winding on arbor, so that at one its side the surface layer was extended, whereas at another side the surface layer was compressed. In order to promote MT, before



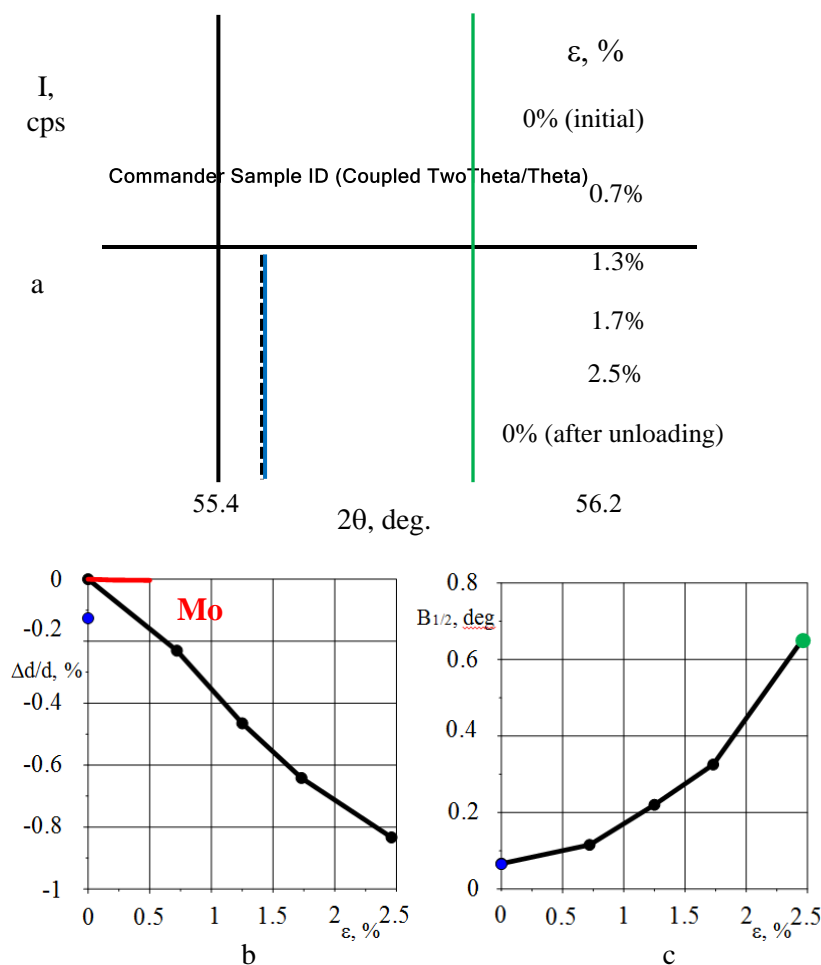
deformation of the foils they were annealed at the recrystallization temperature of 700°C [3], since in perfect, coarse grains martensitic needles move easier. Then winded annealed foils were straightened partially or completely for the sake of convenient X-ray measurements, especially – texture measurements. By straightening in annealed foils MT  $\beta \rightarrow \alpha''$  begins, so that both stages of superelasticity can be observed.

X-ray studies of foil samples were conducted by automated texture diffractometers DRON-3 and D8 DISCOVER of Russian and German (BRUKER) manufacture, respectively. Both diffractometers were used by registration of X-ray line profiles, DRON-3 – by measurement of direct texture pole figures (DPF) [5], D8 DISCOVER – by measurement of generalized pole figures (GPF) [6].

## 2. Experimental results

### 2.1. Stretching of foil

The foil sample was stretched in the special set to diffractometer up to  $\varepsilon=2.5\%$  by continuous registration of the diffraction spectrum using a position-sensitive detector. Under tensile deformation X-ray lines of the initial  $\beta$ -phase shift to higher Bragg angles (Figure 1-a,b) and their profiles become wider (Figure 1-c), but by removal of loading both positions of lines and their angular widths return to their initial values. When taking into account, that the foil is subjected to tension in the rolling plane, it is understandable, that along perpendicular to rolling plane (ND, that is normal direction) material of the foil experiences compression, – namely therefore its diffraction lines shift to higher angles. Since the almost complete recovery of X-ray line parameters occur after deformation degree of 2.5%, it becomes evident, that we deal with superelasticity.

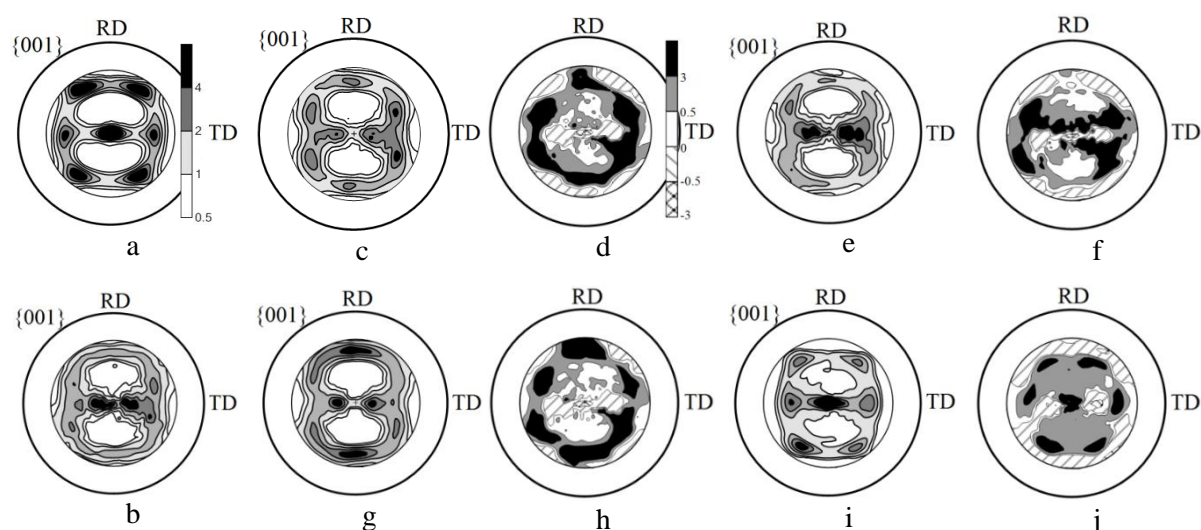


**Figure 1.** Change of line profile (a), interplanar spacing (b) and dispersion of crystal lattice (c) during increasing sample deformation by tension. Green color is used for sign of data on sample with the maximal deformation, blue – for unloaded sample.

Behavior of the foil from TNZ alloy under tension within the limits of elastic deformation was compared with that of the molybdenum foil (Figure 1). The return of X-ray line parameters, i.e. peak position and half-width of the profile  $\beta$ , to their initial values after unloading of samples is the general property of metal materials, though concrete trajectories of  $\beta$  recovery vary in details. Mechanisms of this effect are different in cases of Mo and TNZ alloy: broadening of X-ray lines due to usual elastic deformation is connected with inhomogeneous changes of interplanar spacing in grains of  $\beta$ -phase, whereas broadening of X-ray lines in the course of superelastic deformation is conditioned by spreading of martensitic needles, subdividing  $\beta$ -grains. Recovery of X-ray lines half-width by unloading of sample in cases both of elastic deformation and superelasticity occurs by redistribution of regions with different interplanar spacings or different degrees of  $\beta \leftrightarrow \alpha''$  MT. Finally, the half-width decreases down to its initial value independently on the trajectory of this return, if only irreversible plastic deformation would not prevent it.

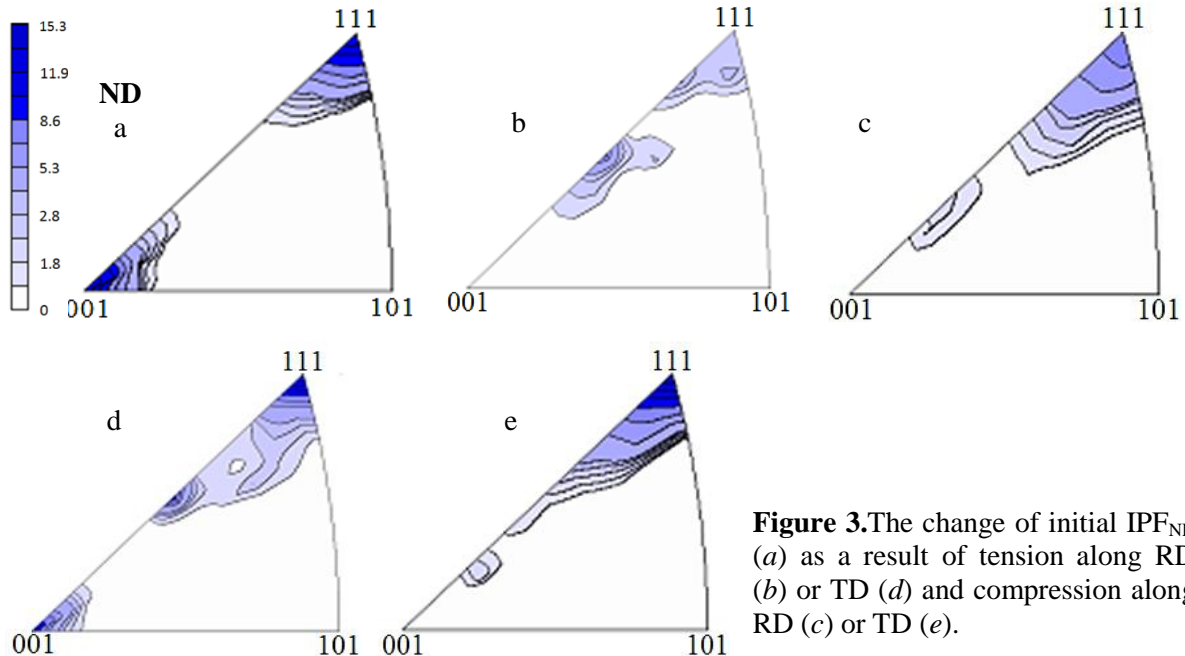
## 2.2. Bending of foil

The samples of foil 17x17 mm in size were wound on the arbor 11 mm in diameter around rolling direction (RD) or transverse direction (TD), annealed at 700°C, straightened and stuck to the plain holder in the predeformed state. At that the deformation degree of the surface layer was equal to ~1.5%. For both sides of straightened foil samples partial DPF {002} with angular radius 70° were measured (Figure 2). In order to see visually changes in DPF due to bending of foils, subtraction pole figures DPF  $\Delta$  were constructed as well. Here  $\Delta(\psi, \phi) = P_{\text{def}}(\psi, \phi) - P_{\text{in}}(\psi, \phi)$ ,  $P_{\text{in}}$  and  $P_{\text{def}}$  – pole densities in points with angular coordinates  $(\psi, \phi)$  of DPF {002} for initial rolled foil after annealing and for straightened foil, annealed after winding. Since shown DPF {002} are normalized [5], the real decrease of pole density in the center of DPF, corresponding to grains with rolling plane {001}, is accompanied with the supposed its increase in points, where pole densities do not change, including those, corresponding to grains with rolling plane {111}. This explains features of the observed distribution in DPF  $\Delta(\psi, \phi)$  (Fig. 2). It is evident, that as a result of MT  $\beta \rightarrow \alpha''$  the volume fraction of  $\beta$ -grains in the texture component with rolling plane {001} decreases, whereas the volume fraction of  $\beta$ -grains in the texture component with rolling plane {111} remains the same or changes insignificantly.



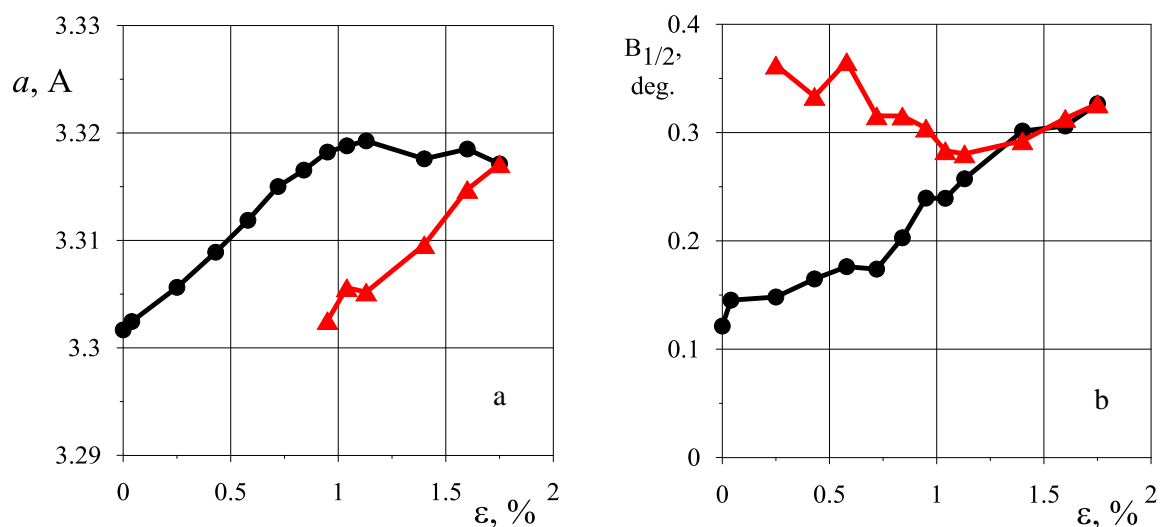
**Figure 2.** Change of texture (PF {001}) of foil from Ti-22%Nb-6%Zr alloy as a result of its bending: a – cold rolling; b – annealing at temperature 700°C during 1 h; c, d – compression along RD (c – PF, d – differential PF); e, f – compression along TD (e – PF, f – differential PF); g, h – tension along RD (g – PF, h – differential PF); i, j – tension along TD (i – PF, j – differential PF).

On the basis of experimental partial DPF{011}, {002} and {112} by use of ODF method [7] with the help of software LABOTEX inverse pole figures (IPF) were obtained for ND, RD and TD of the foil. IPF for ND, shown in Figure 3, confirm data of DPF in Figure 2.

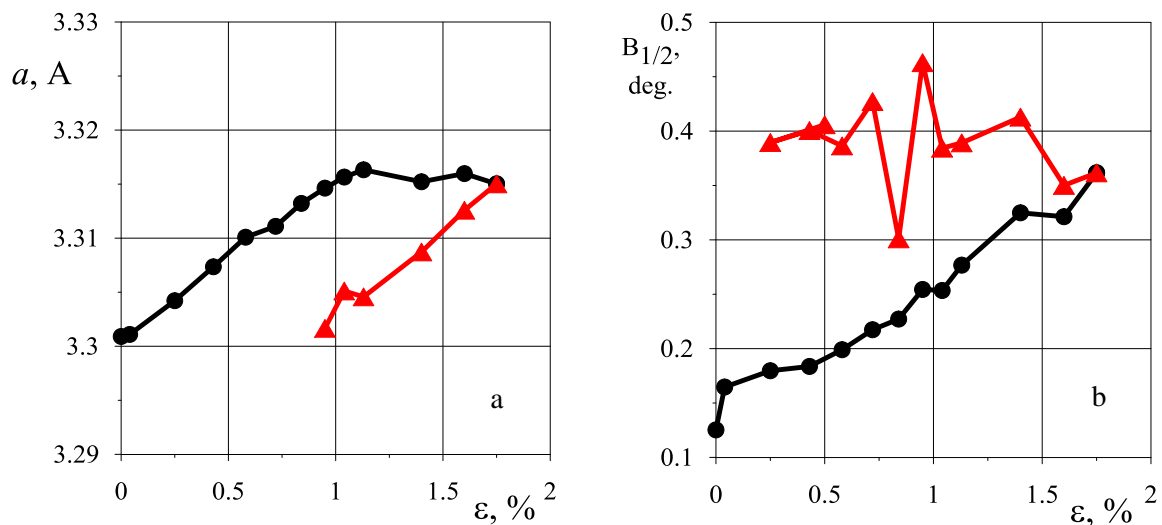


**Figure 3.** The change of initial IPF<sub>ND</sub> (a) as a result of tension along RD (b) or TD (d) and compression along RD (c) or TD (e).

One more experiment consists in gradual straightening of the winded annealed foil by use of the series of arbors with increasing radii; at that, by each radius, corresponding to the definite deformation degree of the surface layer by compression up to 2%, X-ray lines (002) and (222) were registered. As a result of compression along RD the lattice parameter  $a$ , measured along ND, increases as well as angular widths of both X-ray lines. When the increase of the lattice parameter stops due to development of dislocation plastic deformation, the loading was gradually removed and parameter  $a$  returns to the initial value (Figs 4 and 5).



**Figure 4.** Changes of crystal lattice parameter (a) calculated by using of X-ray reflection (200) and half-width of this reflection (b). Black color indicates the process of loading, red – unloading.



**Figure 5.** Changes of crystal lattice parameter (a) calculated by using of X-ray reflection (222) and half-width of this reflection (b). Circle points correspond process of loading, and triangle points – unloading.

The most noteworthy feature of shown curves is the elastic recovery of the lattice parameter  $a$ , whereas the angular half-width of X-ray lines do not show the similar tendency. By unloading of sample the curve  $B_{1/2}(\epsilon)$  for grains with rolling plane  $\{001\}$  coincides with the curve of loading within the interval  $\Delta\epsilon = 1.7 \div 1.3$ , while for grains with rolling plane  $\{111\}$  it deflects from the loading curve starting with the very beginning of unloading. This difference indicates, that in latter grains MT was absent.

### 3. Application of Generalized Pole Figures

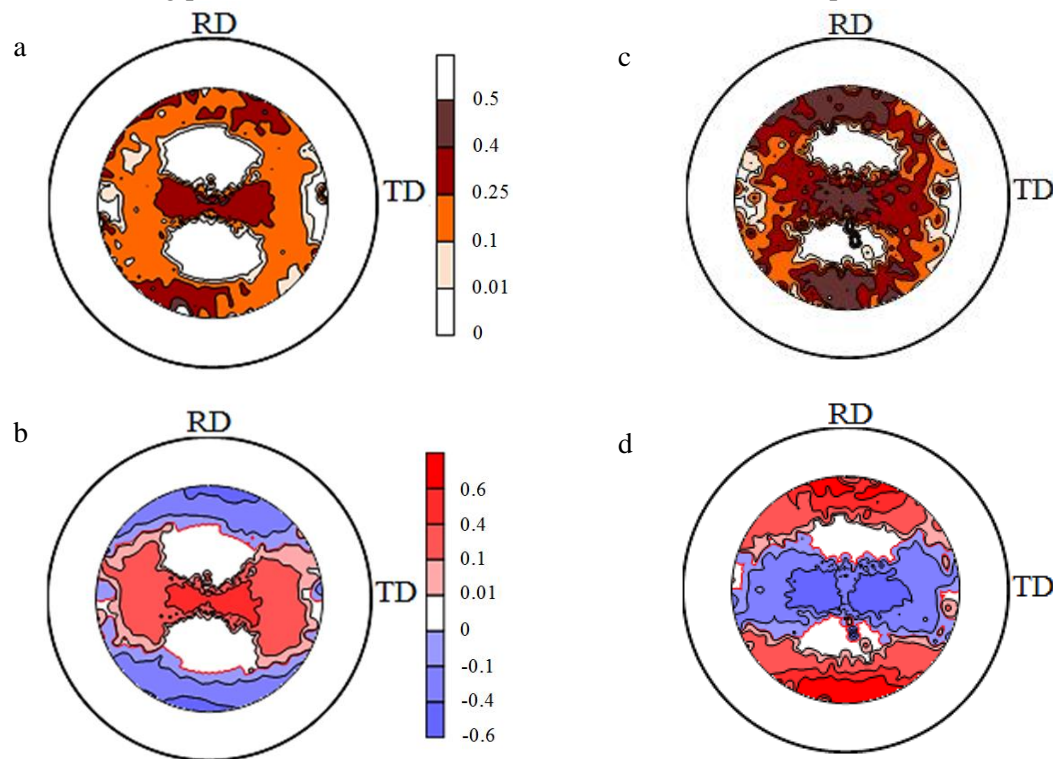
A series of data, confirming the non-uniform character of  $\beta \leftrightarrow \alpha''$  MT in the foil of TNZ alloy, was obtained by the comparatively new X-ray method of Generalized Pole Figures (GPF) [6]. The method involves successive registration of the same X-ray line profile for grains of all orientations in the course of texture measurement using a position sensitive detector. Correction of measured data by use of standard samples and computer treatment of obtained results allowed to construct so called GPF  $\{hkl\}$ , where some measured diffraction parameter or calculated substructure parameter is plotted, so that points  $(\psi, \varphi)$  correspond to reflecting grains with the same coordinates of axes  $\{hkl\}$ .

In particular, in Figure 6 GPF  $\beta_{002}$  and GPF  $(\Delta d/d_{mid})_{002}$  are presented, respectively, for foils, winded along RD. Winded samples were annealed, straightened and fixed on the flat holder. GPF were measured both from compressed and stretched sides of the foil by deformation degree of the surface layer  $\epsilon \approx 1.5\%$ . Presented GPF  $\beta_{002}$  for the compressed side (Figure 6-a) directly confirms, that grains with rolling plane  $\{001\}$ , localized near the center of PF, are characterized with higher strain hardening, i.e. with higher X-ray line broadening  $\beta_{002}$ , than grains with rolling plane  $\{111\}$ , localized at the angular distance of  $55^\circ$  from the center. The reason of this effect is the following. According to the main regularity of texture formation, in rolled BCC-metals grains with rolling plane  $\{001\}$  are larger, than grains with rolling plane  $\{111\}$  [7]. This principle remains true both after recovery and primary recrystallization. Therefore formation of martensitic needles develops in grains with rolling plane  $\{001\}$  more freely, than in grains with rolling plane  $\{111\}$ , without obstacles from grain boundaries.

Since MT  $\beta \rightarrow \alpha''$  occurs with an increase of interplanar spacings [4], stretching of the surface layer promotes this MT, while compression of the surface layer hinders MT. Therefore MT  $\beta \rightarrow \alpha''$  develops more actively on the stretched side of the bend foil, than on its compressed side. As a result, it is



difficult to reveal in GPF regions of preferred passing of MT  $\beta \rightarrow \alpha''$ , though one can suppose, that in grains with rolling plane  $\{001\}$  MT would be most active both under compression and tension.



**Figure 6.** GPF  $\beta_{200}$  (a, c) and GPF  $\Delta d/d_{200}$  (b, d) for the foil sides compressed (a, b) or stretched along RD (c, d).

As for GPF MT  $\beta \rightarrow \alpha''$ , it ought to be noted, that in conditions of compression and tension studied material has a number of resources, realization of which would promote operation of external loading. A stereographic projection of the loaded foil allows to see, how these resources are realized. When, for example, in GPF  $(\Delta d/d_{av})_{002}$  the value  $(\Delta d/d_{av})(\psi, \varphi) > 0$ , it signifies, that in  $\beta$ -grains with axes  $\langle 001 \rangle$ , having coordinates  $(\psi, \varphi)$ , the interplanar spacing  $d$  under applied loading proves to be higher, than its weighted average level, responsible for macrostress. Points  $(\psi, \varphi)$  near the center of GPF correspond to axes  $\langle 001 \rangle$ , directed close to the normal to rolling plane (ND), whereas points in the vicinity of rolling direction (RD) correspond to axes  $\langle 001 \rangle$ , close to RD. Since elastic compression of metal material along some direction is accompanied with its elastic tension in the perpendicular plane and vice versa, the distribution of values  $(\Delta d/d_{av})_{002}$  in the corresponding GPF under uniaxial loading.

As it is seen in Figure 6-b, elastic deformation of  $\beta$ -grains under compression along RD redistributes in such a manner, that along direction of compression in these grains axes  $\langle 001 \rangle$  are situated with values  $\Delta d/d_{av} < 0$ , while across this direction – axes  $\langle 001 \rangle$  with values  $\Delta d/d_{av} > 0$ . Therefore along GPF diameter, perpendicular to direction of compression, the zone, where  $\Delta d/d_{av} > 0$ , is situated.

Along direction of tension in  $\beta$ -grains axes  $\langle 001 \rangle$  with values  $\Delta d/d_{av} > 0$  take up position, while across this direction – axes  $\langle 001 \rangle$  with values  $\Delta d/d_{av} < 0$  (Fig. 6-d). Therefore along GPF diameter, perpendicular to direction of tension, the zone, where  $\Delta d/d_{av} < 0$ , is situated.

Grains of martensitic phase  $\alpha''$  develops in such a manner, that along direction of tension their axes  $\langle 111 \rangle$  with values  $\Delta d/d_{av} > 0$  are situated, while across this direction in GPF  $(\Delta d/d_{av})_{\alpha''}$  – axes  $\langle 111 \rangle$  with values  $\Delta d/d_{av} < 0$  take position.

#### 4. Conclusions

- (1) Superelasticity of Ti-22%Nb-6%Zr alloy is conditioned by passing of elastic deformation of the  $\beta$ -phase lattice into its reversible deformation by  $\beta \rightarrow \alpha''$  MT, beginning from stresses, which are smaller, than the yield point of  $\beta$ -phase.
- (2) An increase of interplanar spacings in the course of  $\beta \rightarrow \alpha''$  MT promotes growth of  $\beta$ -phase elastic deformation by stretching of sample, at least, up to  $\varepsilon=2.5\%$  and impedes elastic deformation of  $\beta$ -phase by compression of the surface layer of sample down to deformation above  $\varepsilon=1\%$ .
- (3) By bending of the foil of TNZ alloy, which, as all metal materials with BCC crystalline lattice, has the two-component rolling texture, when the deformation degree of the surface layer is close to  $\varepsilon=1.5\%$ , MT  $\beta \rightarrow \alpha''$  is localized on the compressed side in less fragmented grains with rolling plane  $\{001\}$ , whereas in grains with rolling plane  $\{111\}$  MT is practically absent.
- (4) On the stretched side of the bent foil MT  $\beta \rightarrow \alpha''$  covers the more significant part of volume and its selectivity is not evident.
- (5) The reversible widening of X-ray lines for samples of superelastic TNZ alloy is conditioned by fragmentation of  $\beta$ -phase grains in the course of MT  $\beta \rightarrow \alpha''$  due to spreading of martensitic needles, whereas their irreversible widening is connected with dislocation plastic deformation; at that, by unloading of sample the reversible part of line widening rehabilitates itself first of all even in the case, when MT  $\beta \rightarrow \alpha''$  is followed by dislocation plastic deformation, since taking down of loading initiates at once MT  $\alpha'' \rightarrow \beta$  and to significant degree removes fragmentation of  $\beta$ -grains, connected with formation of martensitic needles.
- (6) By unloading of the bent foil at its compressed side grains with rolling plane  $\{001\}$  reveal partial narrowing of X-ray line (002) profile, while grains with rolling plane  $\{111\}$  do not show the similar narrowing of line (222), confirming the fact of reversible MT  $\beta \leftrightarrow \alpha''$  in the first group of grains and its absence in the second group.
- (7) It was shown by use of the X-ray method of Generalized Pole Figures, that:
  - by compression in grains with rolling plane  $\{001\}$  strain hardening due to MT  $\beta \rightarrow \alpha''$  is significantly higher, than in grains with rolling plane  $\{111\}$ ;
  - in grains of  $\beta$ - and  $\alpha''$ -phases microdeformation of crystalline lattice in the course of loading redistributes in such a manner, that maximally promotes macrodeformation of the sample;
  - the most intensive MT  $\beta \rightarrow \alpha''$  takes place in grains, showing the greatest tendency to increase the volume of crystalline lattice, i.e. where  $\Delta d/d_{av} > 0$ .

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