

## TRANSFORMATION FEATURES IN Ti-Nb-(Zr,Ta) SMA UNDER VARIABLE STRESS-TEMPERATURE CONDITIONS

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Ti-Nb-based shape memory alloys (SMA) are promising candidates for the role of metallic implant materials because they could be designed to manifest low Young's modulus and superelastic behavior close to that of bone tissues. These alloys are nickel-free, and therefore entirely biocompatible. They are multiphase materials with multiple solid-state phase transformations, however, only one of them, a reversible  $\beta \leftrightarrow \alpha''$  martensitic transformation, leads to shape memory and superelasticity effects. To allow better understanding of the phase transformation phenomena in these alloys under variable stress-temperature conditions, this work is focused on their *in situ* X-ray diffraction analysis.

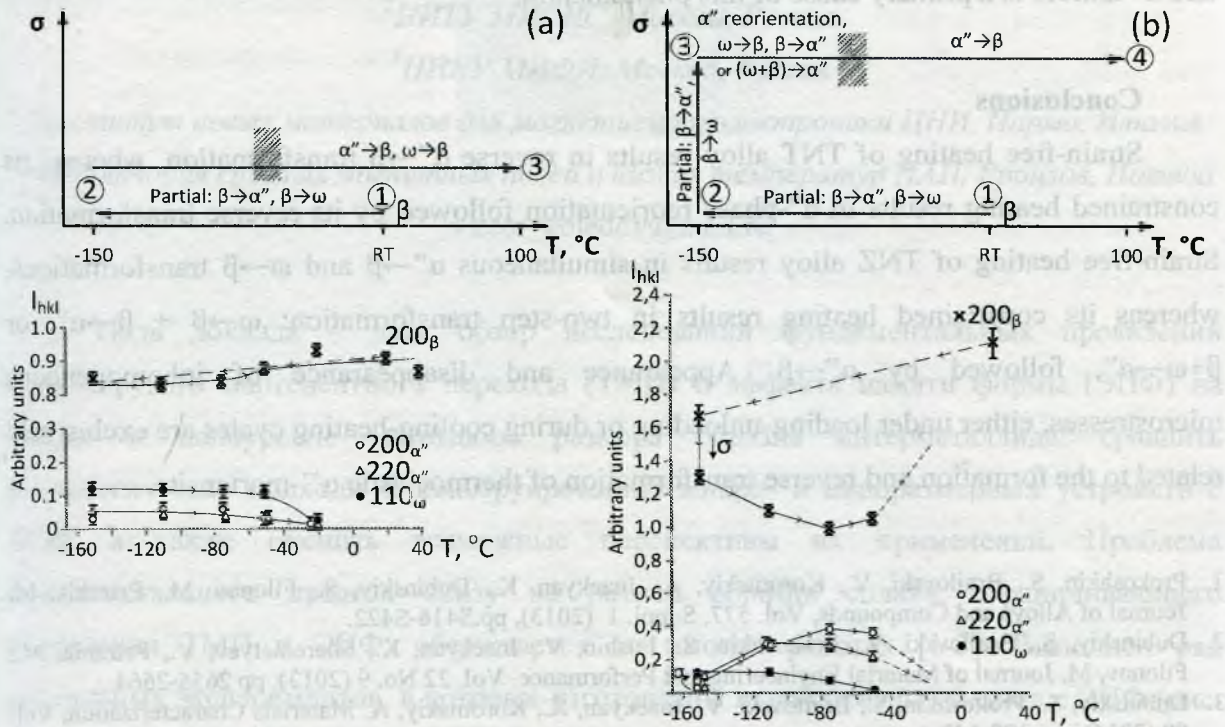
Ti-21.8Nb-6Zr (at.%) and Ti-19.7Nb-5.8Ta (at.%) alloys (TNZ and TNT, respectively) were subjected to cold rolling (true strain  $\epsilon=0.37$ ) and post-deformation annealing (TNZ: 600°C, 30 min; TNT: 500°C, 1h) to create a nanosubgrained structure corresponding to the best combination of functional properties in these alloys [1,2]. TNZ and TNT alloys were chosen for this study due to their different trend to athermal  $\omega$ -phase formation. An original tensile module, which functioning is based on an independent two-way Ti-Ni SMA actuator, lodged within a low-temperature thermal chamber of a diffractometer was used for experiments with and without external load in the -150...+100°C temperature range [3]. Based on the results obtained, analysis of the cooling- and stress-induced  $\alpha''$ - and  $\omega$ -phases direct and reverse transformations in TNZ and TNT alloys is presented in this study.

In both alloys,  $\beta$ -phase is a main phase in the studied temperature range. At RT, the quantity of  $\alpha''$ -phase in TNT is quite small while in TNZ it is hardly detectable. At RT, a small quantities of  $\alpha$ -phase (TNT) and  $\omega$ -phase (TNZ) are also present.

**Ti-Nb-Ta.** Under load-free cooling of TNT from RT to -150°C, the quantity of  $\alpha''$ -phase somewhat increases. Loading at -150°C results in a further increase of the  $\alpha''$ -phase content and in its subsequent reorientation during heating. Loading at RT leads to insignificant changes in  $\alpha''$ -phase quantity, followed by its slight increase during cooling under load. Alpha''-phase reorientation is observed during heating under external load.



**Ti-Nb-Zr.** Cooling of the TNZ results in the appearance of  $\alpha''$ - and  $\omega$ -phase lines despite loading conditions. Upon load-free heating,  $\alpha''$ - and  $\omega$ -phase contents do not change significantly up to  $-75^\circ\text{C}$ , then they rapidly and simultaneously decrease to sink into the background at  $-20^\circ\text{C}$ ; i.e., they show an independent and simultaneous disappearing with temperature increase (Figure 1a).



**Figure 1.** TNZ: X-ray diffraction line integral intensities vs recording temperature without external load (a) and with external load (b) experiments. Shaded areas indicate starting temperatures of the reverse  $\alpha'' \rightarrow \beta$  transformations

Whereas during heating under load the  $\omega$ -phase quantity decreases monotonically, the quantity of  $\beta$ -phase first decreases (up to  $-75^\circ\text{C}$ ) and then increases, while the quantity of  $\alpha''$ -phase shows a mirror to  $\beta$ -phase behavior: it augments up to  $-75^\circ\text{C}$  and then diminishes. Note that at  $-50^\circ\text{C}$ , no  $\omega$ -phase lines can be observed, while the  $200_{\alpha''}$  and  $220_{\alpha''}$  lines are still clearly visible (Figure 1b). Therefore it could be concluded that heating under load of TNZ results in a two-step transformation: (1)  $\omega \rightarrow \beta + \beta \rightarrow \alpha''$ , or  $\beta + \omega \rightarrow \alpha''$  [4] transformations accompanied by  $\alpha''$ -phase reorientation, followed by (2)  $\alpha'' \rightarrow \beta$  transformation.

Under load-free cooling of TNZ and TNT alloys to  $-150^\circ\text{C}$ , a distinct widening of the  $\beta$ -phase X-ray lines accompanies direct  $\beta \rightarrow \alpha''$  transformation. Under heating back to  $100^\circ\text{C}$ , the X-ray line width  $B_{hkl}^\beta$  decrease in the reverse  $\alpha'' \rightarrow \beta$  transformation temperature range down to the initial values. Loading at  $-150^\circ\text{C}$  does not significantly affect  $B_{hkl}^\beta$  values of the already strongly-widened  $\beta$ -phase lines. Upon subsequent heating under load,  $B_{hkl}^\beta$  values decrease down to their initial values as well. Moreover,  $211_{\alpha_1} - 211_{\alpha_2}$  doublet splitting clearly observed



at RT disappears due to strong overlapping of singlets in the temperature range of  $\alpha''$ -phase presence. Consequently, it can definitely be concluded that inhomogeneous microstresses related to the direct  $\beta \rightarrow \alpha''$  (thermally or stress-induced) and reverse  $\alpha'' \rightarrow \beta$  transformations represent the only reasons for the reversible X-ray line width variations. The coherency of  $\beta$  and  $\alpha''$  lattices is a primary cause of this phenomenon.

### Conclusions

Strain-free heating of TNT alloy results in reverse  $\alpha'' \rightarrow \beta$  transformation, whereas its constrained heating results in  $\alpha''$ -phase reorientation followed by its reverse transformation. Strain-free heating of TNZ alloy results in simultaneous  $\alpha'' \rightarrow \beta$  and  $\omega \rightarrow \beta$  transformations, whereas its constrained heating results in two-step transformation:  $\omega \rightarrow \beta + \beta \rightarrow \alpha''$ , or  $\beta + \omega \rightarrow \alpha''$ , followed by  $\alpha'' \rightarrow \beta$ . Appearance and disappearance of inhomogeneous microstresses, either under loading-unloading or during cooling-heating cycles are exclusively related to the formation and reverse transformation of thermoelastic  $\alpha''$ -martensite.

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