THE EFFECT OF COLD ROLLING O MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Ti-35Nb-6Ta ALLOY

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Abstract

The beta titanium alloys are widely used for hard tissue replacements due to their properties (e.g. high strength, low Young's modulus or biocompatibility). Mechanical properties are dependent on microstructure which can be controlled through suitable thermo-mechanical treatment. The effect of cold rolling on texture evolution and mechanical properties has been studied in this paper. Hot forged, solution treated (850 °C / 0.5 h / water quenched) Ti-35Nb-6Ta alloy has been cold rolled with thickness reduction 40, 60 and 80 and 95%. The texture evolution has been studied by using X-ray diffraction analysis and EBSD analysis. The {100} <110> texture is dominant for all studied specimens and became more distinct with increasing thickness reduction. Another texture evolution from {211} <110> to {111} <112> was observed with increasing thickness reduction. The tensile strength increased with thickness reduction from 600 to 830 MPa. On the other hand elongation decreased from 32 to 9%. The Young's modulus value has been determined within range of 43 to 55 GPa.

Keywords: Beta-titanium alloys, mechanical properties, texture, cold rolling

1. INTRODUCTION

The importance of development of new β-titanium alloys for implants is growing simultaneously with increasing demand on hard tissue replacements. The materials for new implants should posses better (not only) mechanical properties the materials that are used currently (e.g. stainless steel, Ti-6Al-4V) [1-2]. The Ti-35Nb-6Ta alloy has been developed for medical applications and contains exclusively fully biocompatible elements. Solution treated alloy posses quite low tensile strength and low Young's modulus. There are several possibilities of improving mechanical properties of β-titanium alloys. Increasing the strength through aging treatment leads to significant increase in modulus [3-4] and therefore another way to material with higher tensile strength and low modulus should be find. Cold deformation seems to be efficient in increasing ultimate tensile strength due to dislocation density increase. The requirements of β-titanium alloys used as materials for implants are numerous (e.g. high strength, corrosion resistance, biocompatibility, low Young's modulus). Desirably high strength to modulus ratio is very difficult to achieve. It has been reported that properties of β-titanium alloys depend on crystallographic directions [5]. Cold rolling could be used to establish desired deformation texture and therefore improve the properties. The lowest Young's modulus in β-titanium (bcc) has been observed in <100> direction whereas the <111> posses the highest modulus value [2, 5 ,6]. The texture development depending on thickness reduction and its correlation has been studied in this work in order to the best process parameters with respect to mechanical properties.

2. METHODS

The Ti-35Nb-6Ta alloy (all compositions in this work are in wt.% unless it said different) has been prepared in a vacuum arc melting furnace LEYBOLD HERAEUS L200h with a water cooled copper crucible. The oxygen content was measured to be between 0.05 and 0.1 %. As-cast specimens were homogenized 1000 °C / 6 h in a vacuum furnace. These specimens were machined and subsequently hot rolled at 750-1050 °C into a rectangular shape rod. The section reduction during hot forging was about 40%. Hot rolled specimens were annealed at 850 °C for 0.5 h and water quenched. The rods were machined in order to remove the oxidized surface layer and defects. Cold rolling with various thickness reductions (i.e. 40, 60, 80 and 95 %) has been performed on specimens. The microstructure has been studied by using light microscopy (LM). Specimens were prepared via standard metallographic route. Specimens were grinded up to #4000 with SiC papers and polished with Struers OP-S emulsion with addition of 0.6 ml OP-S, 2 ml H_2O and 2 ml NH_3. For etching 3 ml HF + 8 ml HNO_3 + 100 ml H_2O etchant was used. Nikon EPIPHOT 300 was used for light microscopy observation. For etching 3 ml HF + 8 ml HNO_3 + 100 ml H_2O etchant has been used. Nikon EPIPHOT 300 was used for light microscopy observation. Polished specimens were used for electron back scattered (EBSD) analysis. For this purpose the specimens were electro-polished by using 1000 ml C_2H_5OH + 50 ml $HClO_4$ + 15 ml HNO_3 electrolyte at 50V. Analysis was performed on JEOL JSM 7600F with EBSD detector (Nordly's - Oxford instruments). EBSD data were collected at 20 kV and 65° tilt. Results were evaluated with HKL Chanel 5 software equipment. Texture was also characterized by X-ray diffraction analysis (XRD). XRD analysis was carried out on vertical θ/θ X'Pert PRO MPD diffractometer using Schultz reflection geometry and radiation from X-ray tube with copper anode (λ = 0.15419 nm). Textures are noted as plane {hkl} parallel to rolled surface (NL) and direction <hkl> parallel to rolling direction (RD). Mechanical properties (except of specimen rolled with 95% thickness reduction) were obtained from tensile tests of "dog-bone" shaped flat specimens on INSTRON 1185 testing machine equipped with video-extensometer. The dimensions of tested specimens were 3 mm in width and 0.9 - 4 mm thickness. The gauge length was 20 mm. Specimens were tested in the direction parallel to rolling direction (RD). At least three specimens were tested for each value. Vickers hardness tests (HV10) were carried out on Zwick Roell ZHU 250 Top testing machine.

3. RESULTS AND DISCUSSION

The microstructure of as-cast and homogenized alloy consists of very coarse and elongated grains. The microstructure has been significantly refined due to dynamic and post-dynamic recrystallization during hot rolling and subsequent solution treatment. It can be seen in **Fig. 1**, where the microstructure of solution treated specimen is presented, that the microstructure consists of relatively fine (~100 - 200 µm) equiaxed

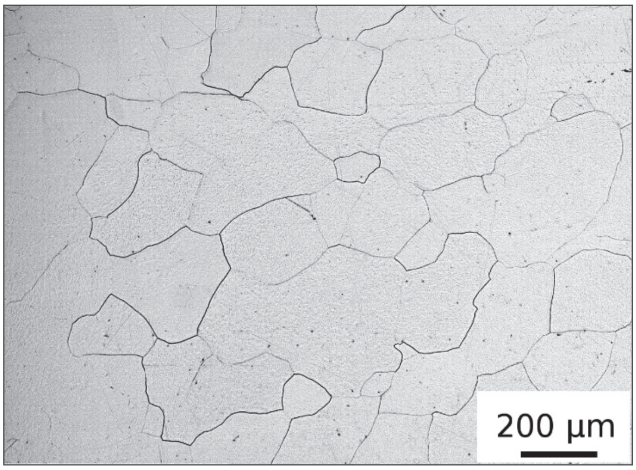

Fig. 1 The microstructure of solution treated Ti-35Nb-6Ta alloy

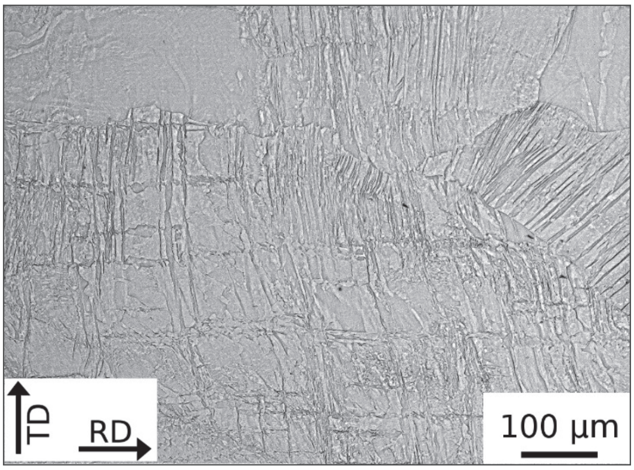

Fig. 2 The microstructure of Ti-35Nb-6Ta alloy cold rolled with 40% thickness reduction

β-grains. Not all grains after solution treatment are fully recrystallized and locally coarser deformed grains can be observed. Weak η-fibre texture (<100> parallel to hot rolling direction) can be distinguished in solution treated alloy, but this texture is not strong. The formation of η-fibre texture in hot rolled β-titanium alloys was also observed by Sander et al. [7].

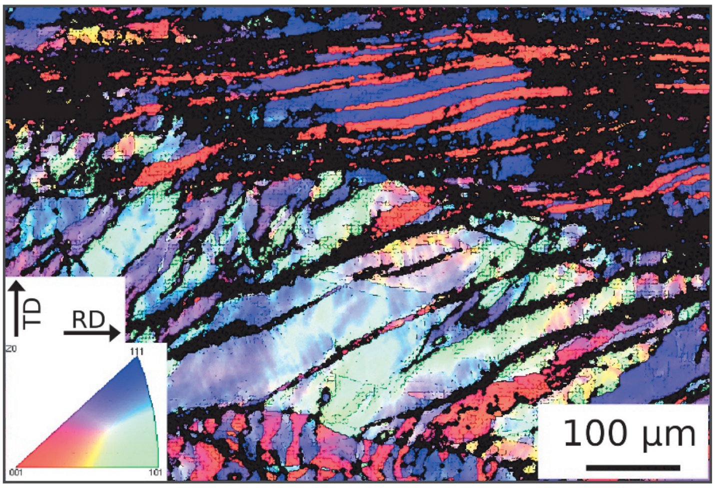

Fig. 3a EBSD of 60% deformed alloy with twins and deformation bands

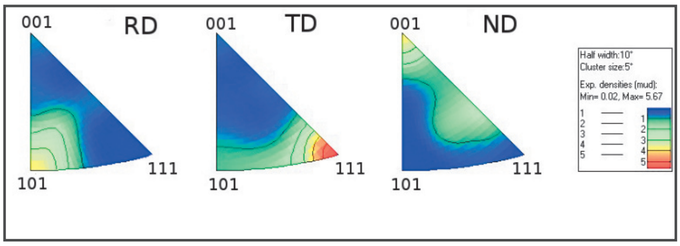

Fig. 3b Inverse pole figures of 60% deformed specimen (obtained by EBSD) showing the dominant {100} <110> texture and weaker {211} <110> texture

The microstructure after cold rolling exhibits elongated original grains in the rolling direction (RD) - **Fig. 2**. Deformation bands or twins can be observed inside these grains (see **Fig. 3**). The twins were analyzed to be in general {112} <111> β twins (CSL = 3) [8, 9], which are typical for β-titanium alloys with low β-phase stability [10 - 12]. Also deformation bands (with various misorientations) have been observed. Those effects are more clearly visible in specimens with lower thickness reduction (40 or 60%) as they are in general quite fine in specimens with higher thickness reductions (i.e. 80%).

The texture analysis revealed that even after 40% thickness reduction some preferred orientations has been established (see **Fig. 4a**), however they are not well developed. Weak residual η-fibre texture can be observed along with other textures (e.g. {111} <112>). In specimen with 60% (**Figs. 3b** and **4b**) reduction {100} <110> and also other textures (e.g. {211} <110>) can be distinguished. With increasing thickness reduction the {100} <110> became dominant (**Figs. 4c, d**) - the 80% and 95% deformed specimens posses strong {100} <110> texture (stronger in 95% deformed specimen).

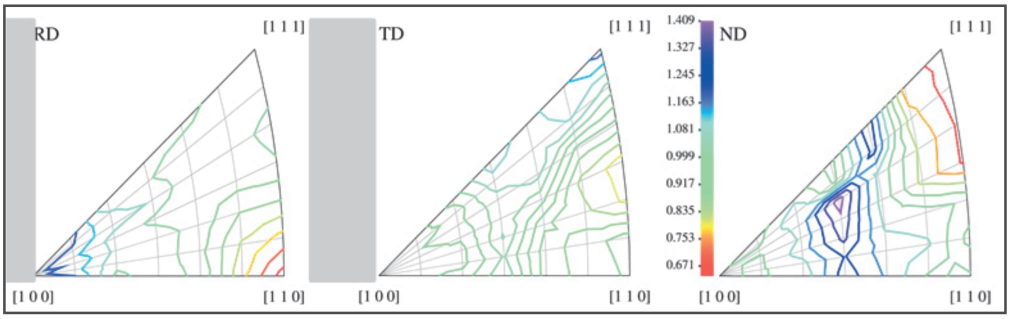

Fig. 4a Inverse pole figures of 40% deformed specimen (obtained by XRD)

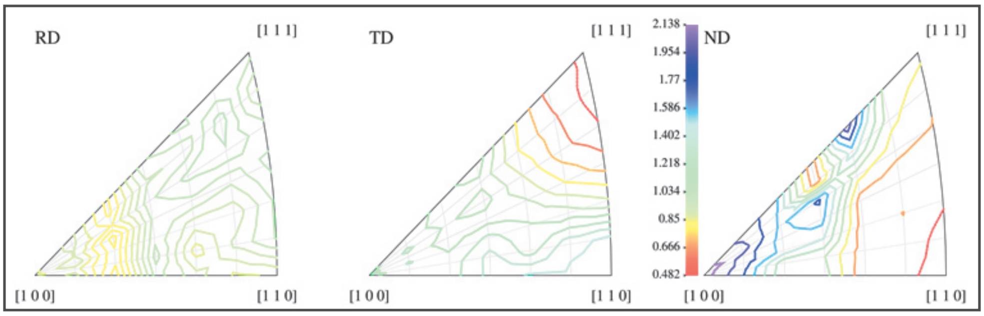

Fig. 4b Inverse pole figures of 60% deformed specimen (obtained by XRD)

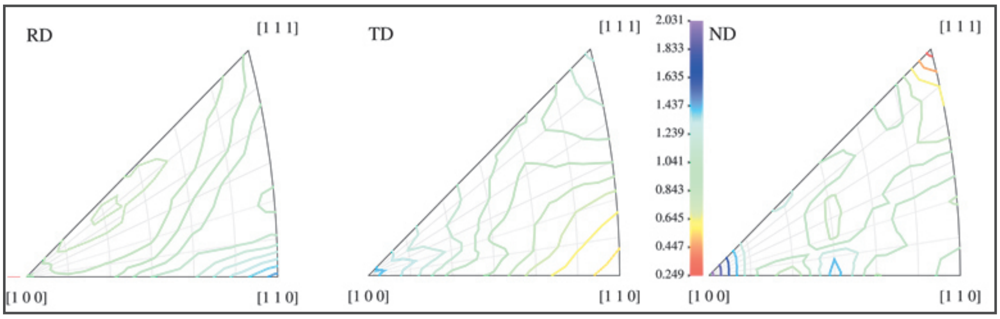

Fig. 4c Inverse pole figures of 80% deformed specimen (obtained by XRD)

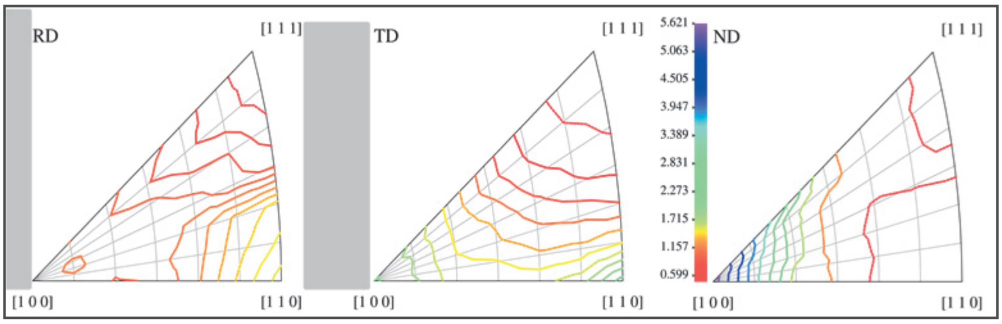

Fig. 4d Inverse pole figures of 95% deformed specimen (obtained by XRD)

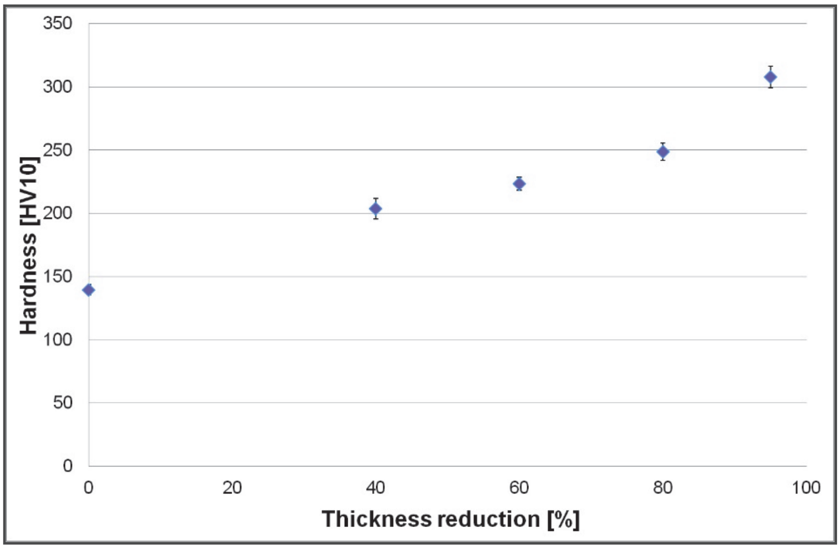

Fig. 5 Hardness vs. thickness reduction

Mechanical properties of cold rolled alloy were studied in direction parallel to RD. The results of tensile tests and hardness tests are plotted in **Fig. 5** and **Fig. 6** respectively. The hardness (**Fig. 6a**) increased from 140 HV10 to more than 300 HV10 due to deformation strengthening. It is also evident that both the ultimate tensile strength (R_m) and 0.2 proof strengths ($R_{p0.2}$) significantly increased with increasing cold deformation. The tensile strength increased from 600 MPa in solution treated specimens to 830 MPa of specimen deformed with 80% thickness reduction. The same situation is for the yield strength values (from 385 to 720 MPa). The deformation strengthening has significant influence on this alloy and it is linearly dependent on thickness reduction within studied thickness reduction range. Elongation values of specimens decreased simultaneously with tensile strength (or deformation) increase.

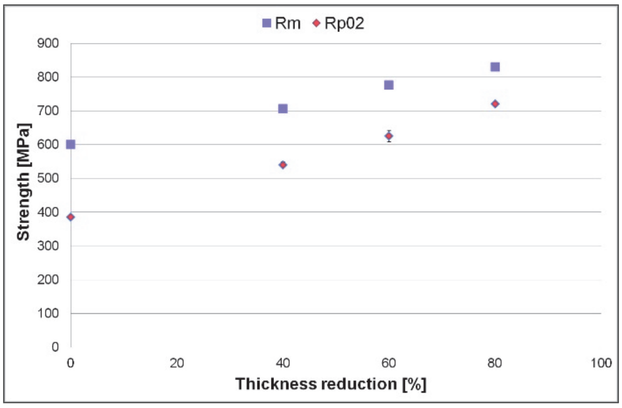

Fig. 6a Tensile strength vs. thickness reduction

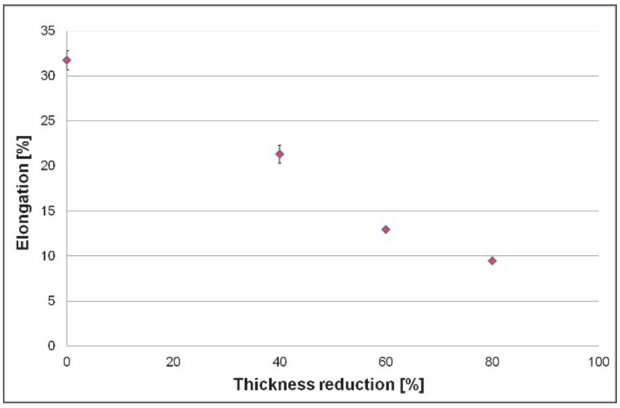

Fig. 6b Elongation vs. thickness reduction

Young's modulus values decreased significantly from 55 GPa to values less than 45 GPa. It has been reported [6, 13, 14] that the lowest Young's modulus in β-titanium is in <100> direction. The modulus is slightly higher in <110> whereas <111> direction posses the highest modulus along to theoretical calculations. Therefore it can be assumed that the decrease in modulus is to a certain amount caused due to {001} <110> texture evolution during cold rolling. Similar texture obtained by cold rolling was observed in Ti-Nb-Al alloy by Inamura et al. [15]. It means that the <110> direction (with small Young's modulus) is preferentially oriented in rolling direction which was parallel to tensile test axis direction. In specimens with lower thickness reductions more grains are oriented with other directions (with higher modulus) parallel to tensile test axis.

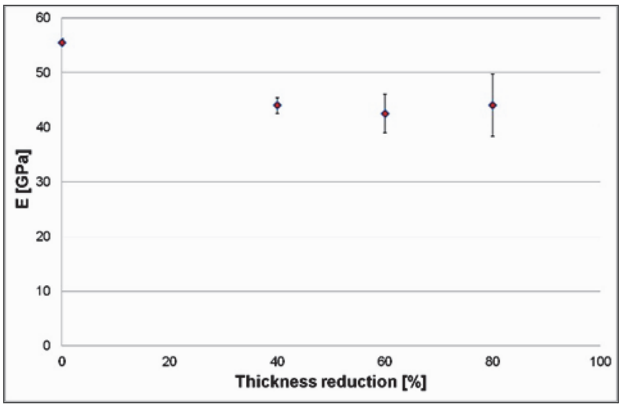

Fig. 6c Young's modulus vs. thickness reduction

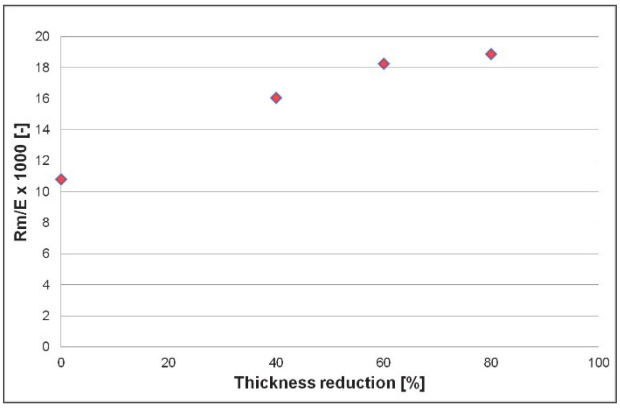

Fig. 6d Strength to modulus ratio vs. thickness reduction

4. CONCLUSIONS

Ti-35Nb-6Ta alloy was processed by hot rolling, solution treatment and cold rolling with various thickness reduction. The texture evolution and correlating mechanical properties have been studied. It can be concluded that:

- The {100} <110> texture is stronger with increasing thickness reduction. In 95% deformed specimen this texture is dominant. Other textures are significant in specimens with lower thickness reduction.
- The strength to modulus ration increased with increasing thickness reduction as the result of increasing strength (from 600 to 830 MPa) and decreasing Young's modulus (from 55 to 45 GPa). The former increased due to deformation strengthening and the latter due to texture development.

ACKNOWLEDGEMENTS

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REFERENCES

[1] DAI S., WANG Y., CHEN F., YU X., ZHANG Y. Effects of cold deformation on microstructure and mechanical properties of Ti-35Nb-9Zr-6Mo-4Sn alloy for biomedical applications. Mater. Sci. Eng. A, Vol. 575, 2013, pp. 35-40.

[2] NIINOMI M., NAKAI M., HIEDA J. Development of new metallic alloys for biomedical applications. Acta Biomater., Vol. 8, No. 11, 2012, pp. 3888-3903.

[3] NAKAI M., NIINOMI M., AKAHORI T., TSUTSUMI H., OGAWA M. Effect of oxygen content on microstructure and mechanical properties of biomedical Ti-29Nb-13Ta-4.6Zr alloy under solutionized and aged conditions. Mater. Trans., Vol. 50, No. 12, 2009, pp. 2716-2720.

[4] FERRANDINI P.L., CARDOSO F.F., SOUZA S.A., AFONSO C.R., CARAM R. Aging response of the Ti-35Nb-7Zr-5Ta and Ti-35Nb-7Ta alloys," J. Alloys Compd., Vol. 433, No. 1-2, 2007, pp. 207-210.

[5] TOBE H., KIM H.Y., MIYAZAKI S. Effect of Nb content on deformation textures and mechanical properties of Ti-18Zr-Nb biomedical alloys. Nippon Kinzoku Gakkaishi/Journal Japan Inst. Met., Vol. 72, No. 12, 2008, pp. 965-969.

[6] ZHANG Y.W., LI S.J., OBBARD E.G., WANG H., WANG S.C., HAO Y.L., YANG R. Elastic properties of Ti-24Nb-4Zr-8Sn single crystals with bcc crystal structure. Acta Mater., Vol. 59, No. 8, 2011, pp. 3081-3090.

[7] SANDER, B. RAABE D. Texture inhomogeneity in a Ti-Nb-based β-titanium alloy after warm rolling and recrystallization. Mater. Sci. Eng. A, Vol. 479, No. 1-2, 2008, pp. 236-247.

[8] BESSE M., CASTANY P., GLORIANT T. Mechanisms of deformation in gum metal TNTZ-O and TNTZ titanium alloys: A comparative study on the oxygen influence. Acta Mater., Vol. 59, No. 15, 2011, pp. 5982-5988.

[9] BERTRAND E., CASTANY P., PÉRON I., GLORIANT T. Twinning system selection in a metastable β-titanium alloy by Schmid factor analysis. Scr. Mater., Vol. 64, No. 12, 2011, pp. 1110-1113.

[10] LI Q., NIINOMI M., NAKAI M., CUI Z., ZHU S., YANG X. Effect of Zr on super-elasticity and mechanical properties of Ti-24at% Nb-(0, 2, 4)at% Zr alloy subjected to aging treatment. Mater. Sci. Eng. A, Vol. 536, 2012, pp. 197-206.

[11] SUN F., ZHANG J.Y., MARTELEUR M., GLORIANT T., VERMAUT P., LAILLÉ D., CASTANY P., CURFS C., JACQUES P.J., PRIMA F. Investigation of early stage deformation mechanisms in a metastable β titanium alloy showing combined twinning-induced plasticity and transformation-induced plasticity effects. Acta Mater., Vol. 61, No. 17, 2013, pp. 6406-6417.

[12] FAN J.K., KOU H.C., LAI M.J., TANG B., CHANG H., LI J.S. Hot deformation mechanism and microstructure evolution of a new near β titanium alloy. Mater. Sci. Eng. A, Vol. 584, 2013, pp. 121-132.

[13] FURUTA T., KURAMOTO S., HWANG J., NISHINO K., SAITO T. Elastic deformation behavior of multi-functional Ti-Nb-Ta-Zr-O alloys. Mater. Trans., Vol. 46, No. 12, 2005, pp. 3001-3007.

[14] DANNO A., WONG C.C., TONG S., JARFORS A., NISHINO K., FURUTA T. Effect of cold severe deformation by multi directional forging on elastic modulus of multi functional Ti+25mol% (Ta,Nb,V)+(Zr,Hr,O) alloy. Mater. Des., Vol. 31, No. Suppl. 1, 2010, pp. S61-S65.

[15] INAMURA T., FUKUI Y., HOSODA H., WAKASHIMA K., MIYAZAKI S. Relationship between texture and macroscopic transformation strain in severely cold-rolled Ti-Nb-Al superelastic alloy. Mater. Trans., Vol. 45, No. 4, 2004, pp. 1083-1089.