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Micro-hardness and Young's modulus of a thermo-mechanically processed biomedical titanium alloy

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Abstract. This paper presents a study on the influence of different thermo-mechanical processing (TMP) parameters on some required properties such as micro-hardness and Young's modulus of a novel near β alloy Ti-20.6Nb-13.6Zr-0.5V (TNZV). The TMP scheme comprises of hot working above and below β phase, solutionizing treatment above and below β phase coupled with different cooling rates. Factorial design of experiment is used to systematically collect data for micro-hardness and Young's modulus. Validity of assumptions related to the collected data is checked through several diagnostic tests. The analysis of variance (ANOVA) is used to determine the significance of the main and interaction effects. Finally, optimization of the TMP process parameters is also done to achieve optimum values of the micro-hardness and Young's modulus.

Keywords: titanium alloys; biomedical applications; micro-hardness; Youngs's modulus; ANOVA

1. Introduction

Titanium (Ti) and its alloys are considered the best choice for replacing or repairing failed hard tissues (structural biomedical applications) as these materials present excellent biocompatibility, high strength to density ratio, outstanding corrosion resistance as well as low modulus of elasticity (Wang 1996, Long *et al.* 1998, Niinomi 2003).

Multifunctional β-type Ti alloys which widely used in various biomedical applications have been developed all over the world. Recently, some new metastable β-type Ti alloys containing β-stabilizers such as Nb and Zr have attracted much special attention for orthopedic implants applications owing to their unique combination of better mechanical properties, low elastic modulus, superior biocorrosion resistance, nontoxicity against osteoblastic cells, no allergic problems, and excellent biocompatibility. The required mechanical properties in this kind of Ti alloys can be improved due to solid solution and second phase strengthening while preserving the

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light weight characteristics of Ti (Karthega *et al.* 2007, Raabe *et al.* 2007). From crystallographic insight, the body centered cubic structure (bcc) of β phase shows higher symmetry as compared to the hexagonal closed packing (hcp) α phase resulting in an isotropic mechanical behavior. Moreover, It is found that their elastic modulus can be significantly reduced by adjusting the concentration of β stabilizing elements (Niinomi 2002, Ikehata *et al.* 2004, Abdel-Hady *et al.* 2006) which makes them appropriate for load bearing surgical implants. Therefore, Ti-based alloys with nontoxic and non-allergic elements such as Nb, Zr, and other elements have been widely used to design new β-type Ti alloys (Been and Grauman 2000). In this regard, the addition of Nb and Zr is preferable to develop absolutely safe Ti-based alloys for biomedical applications depending upon its ability to achieve biological passivity and capacity of reducing the elastic modulus (Yang and Zhang 2005).

In general, thermo-mechanical processing (TMP) is a metallurgical process that integrates work hardening and heat-treatment into a single process (Degarmo *et al.* 2003) plays a crucial role to produce a microstructure with outstanding properties in the materials (Weaver and Garmestani 1998, Bache and Evans 2001, Lonardelli *et al.* 2007). The mechanical properties depend strongly on the alloy composition, processing history, heat treatment conditions which decide the varieties of microstructures (Ding *et al.* 2002). Near-β Ti alloys respond to thermal treatment and TMP and various microstructural constituents like the size, shape and the amount of the various phases can be modified by varying the TMP parameters. However, the influence of thermal treatment and TMP on microstructural features of as-cast Ti-Nb-Zr alloy system and in turn on its mechanical is scarcely reported. Majumdar et al. studied the role of TMP on microstructure and mechanical properties of Ti-13Nb-13Zr alloy (TNZ) (Majumdar *et al.* 2011). It is found that the major results were mainly depending upon the cooling rate after solution treatment. The presence of high amount of α phase in the microstructure showed high Young's modulus whereas the presence of α'' martensite and retained β phases lowered the modulus of the samples.

Nowadays, great efforts in terms of extensive work and focus are being dedicated by engineers and materials scientists in developing novel Ti alloys for biomedical applications with superior mechanical properties and low elastic modulus. In this study, titanium-niobium (Nb)-zirconium (Zr) based alloy containing small amounts of vanadium (V) was investigated in order to evaluate its possible application as a biomedical material. Nb, Zr and V, having a β-phase stabilizing effect for Ti materials, were chosen to control microstructure desirably. In spite of the fact V is associated with toxicity, the concentration used in this study is minimal. Microstructure control was carried out by performing hot working in β phase field as well as (α+β) field subsequently heat treatment at β phase field and also at (α+β) field in different cooling rates. Analysis of Variance (ANOVA) is one of the most frequently used statistical techniques which is used to evaluate the effect of TMP parameters on the requisite properties of biomedical Ti alloys. It appears from the reported literature that very few researchers have applied the statistical technique to investigate the effect of TMP parameters on the required properties of biomedical Ti alloys. This paper has made an attempt to statistically analyze the effect of TMP parameters on some essential properties of a new TNZV alloy such as micro-hardness (HV) and Young's modulus (E). The results are explained with the help of firm technical discussions derived and co-related to micro-structure.

2. Experimental procedure

The alloy in the present investigation was cast using the facility available at DMRL, India with mixture of sponge Ti along with Nb powder and Zr chips as raw materials. The Ti-20.6Nb-13.6Zr-0.5 V (TNZV) alloy was prepared using the non-consumable vacuum arc melting technique and supplied in the form of 600g pancakes. The pancakes were re-melted three times to ensure compositional homogeneity, so obtaining compact and homogenous ingots with neither weight loss nor oxidation. The composition of the alloy was analyzed and the same in wt% is given in Table 1.

The as-cast TNZV alloy was then heat-treated at 1000℃ for 1h for homogenization, and water cooled. In order to determine the β transition temperature (β transus) of the newly developed TNZV alloy, differential scanning calorimeter (DSC) analyses have been performed under protective argon atmosphere, at a scanning rate of 10℃/min, to the maximum temperature of 800℃. The β transus of the alloy was measured to be 695℃. Subsequently, the homogenized samples were given 10% reduction by forging at above (850℃) and below (650℃) the β transition temperature and directly subjected to 25% reduction by rolling at same temperatures and were then air cooled to room temperature. After entire plastic working was accomplished, the alloy remained free from any of the metal working defects which indicated that the entire metal working process was performed successfully. The hot deformed TNZV samples were also solution treated at 850℃ (above β-transus) and 650℃ (below β-transus) for 1h in a dynamic argon atmosphere; this was followed by furnace cooling (FC), air cooling (AC), and water quenching (WQ). The entire TMP scheme of TNZV alloy is shown in Fig. 1.

The composition of the major and trace elements was determined using X-Ray Fluorescent (XRF) Spectrometer (Oxford- X Srata, model: ISIS 1559). Microstructure analysis of the heat treated samples was carried out using an optical microscope (Carl Zeiss with Clemax Software version 3) and field emission scanning electron microscope (FE-SEM, NOVA NANO SEM 450 FEI, Netherlands) at 2 kV. For this, the metallographic samples were prepared using standard techniques for Ti and its alloys (Kuhn and Medlin 1972). The samples were ground to 1200 grit with silicon carbide (SiC), followed by final polishing to a mirror finish using 0.5 μm diamond paste. The metallographically polished samples were etched with Kroll's reagent (10 vol% HF and 5 vol% HNO_3 in water). Room temperature X-ray diffraction analysis was carried out using an X-ray Diffractometer, Philips, Holland, PW 1830 with Cu Kα radiation (wavelength 1.54056°A) at an accelerating voltage of 40 kV and a current of 30 mA.

Vickers micro-hardness measurements were performed using a computer controlled precision micro-hardness tester (model: MicroWhizHard; make: Mitutoyo, Japan) with an indentation load of 300 gf and a dwell time of 5 seconds for each of the indents. Ten indentations were taken for each specimen and the average values were considered. Hardness measurements were carried out on the samples finished using 0.5 μm diamond paste.

Tensile testing was performed as per ASTM E8M to determine Young's modulus of all heat treated samples using a conventional tensile testing unit (Computerized FIE Make Universal Testing Machine, Model UTE-60), with a constant cross-head speed of 1 mm/min in air at room temperature.

The Young's modulus (E) was obtained by measuring the slope of the linear part of stress-strain response. Dog-bone-shaped tensile specimens with dimensions: 10 mm width, 4 mm in thickness and a gage length of 25 mm were precisely machined using Wire Electrical Discharge Machine (Wire-EDM). After machining, tensile specimens were polished using SiC waterproof papers of up to #2500 grit and the gage length of the specimens was mechanically polished using a diamond paste with a particle size 0.5 μm.

Table 1 The chemical composition (wt%) of TNZV alloy used in this study

Ti (wt %)	Nb (wt %)	Zr (wt %)	V (wt %)	Fe (wt %)
Balance	20.6	13.6	0.5	1.4

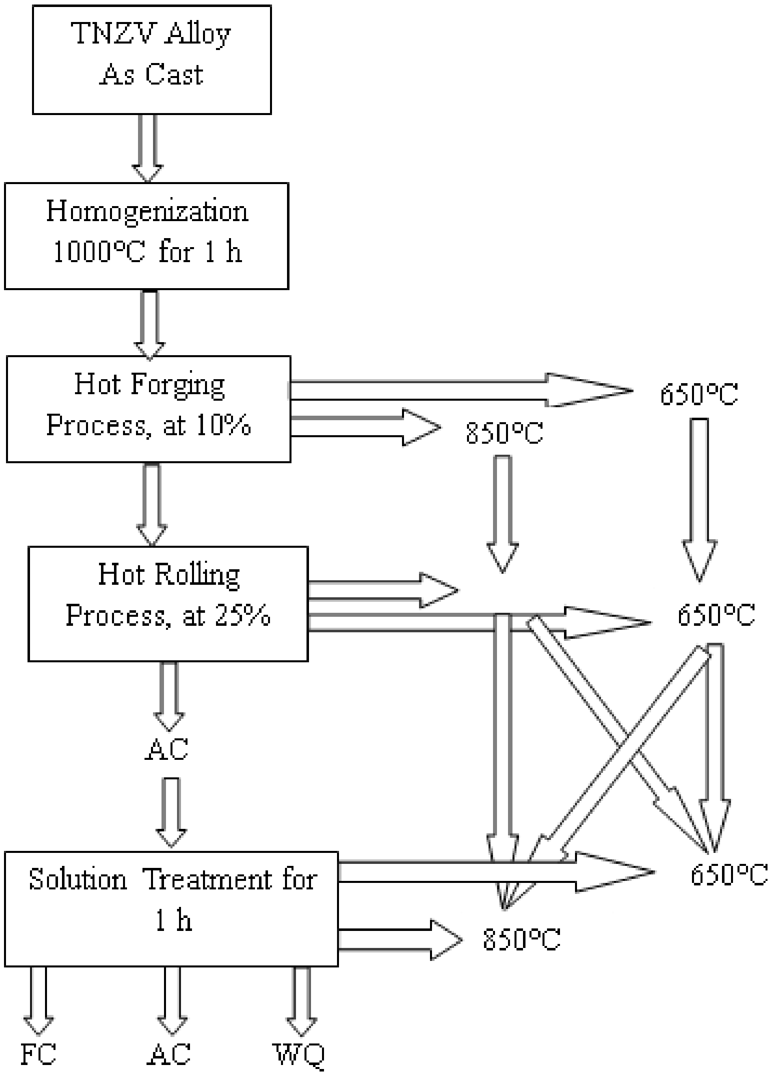


Fig. 1 Flow chart depicting the schedule for Thermo-mechanical processing

3. Design of experiments

In this study, three TMP parameters i.e., hot working temperature, solution treatment temperature and type of cooling were considered. Two levels each for working temperature and solution treatment temperature and three levels of type of cooling were considered. Table 2 shows the TMP parameters and their levels.

Table 2 Experimental factors and their levels

Factor	Symbol	Unit	Level-1	Level-2	Level-3
Working temperature	*A*	℃	650	850	
Solution treatment temperature	*B*	℃	650	850	
Cooling type	*C*		Furnace cooling (FC)	Air cooling (AC)	Water quenching (WQ)

Table 3 Experimental results of HV and E

Run	Working temperature	Solution treatment temperature	Cooling type	HV	E (GPa)
1	850℃	850℃	WQ	222	59
2	650℃	850℃	FC	231	79
3	650℃	650℃	AC	263	77
4	650℃	650℃	WQ	230	75
5	850℃	850℃	AC	239	76
6	650℃	650℃	FC	240	79
7	650℃	850℃	AC	244	78
8	850℃	650℃	WQ	260	77
9	850℃	850℃	FC	236	80
10	850℃	650℃	FC	268	81
11	650℃	850℃	WQ	217	63
12	850℃	650℃	AC	279	79

4. Results and discussion

4.1 Microstructural analysis of as Cast TNZV Alloy

The microstructure observations of the as-cast present TNZV alloy which is called as "metastable-β- alloy" performed using optical microscopy (OM) and SEM. These microstructural tests showed the presence of fine needle like α phase (acicular α) in former β-phase matrix with segregation of α phase on grain boundaries (Fig. 2(a)-(b)). For each former β-grain, twelve different crystal orientations of the α precipitates (variants) are available according to the Burgers relationship (Burgers 1934), which links particular crystal directions and planes of both phases

$$\langle 111\rangle_{\beta} \,//\, \langle 1120\rangle_{\alpha},\ (110)_{\beta} \,//(0001)_{\alpha}$$

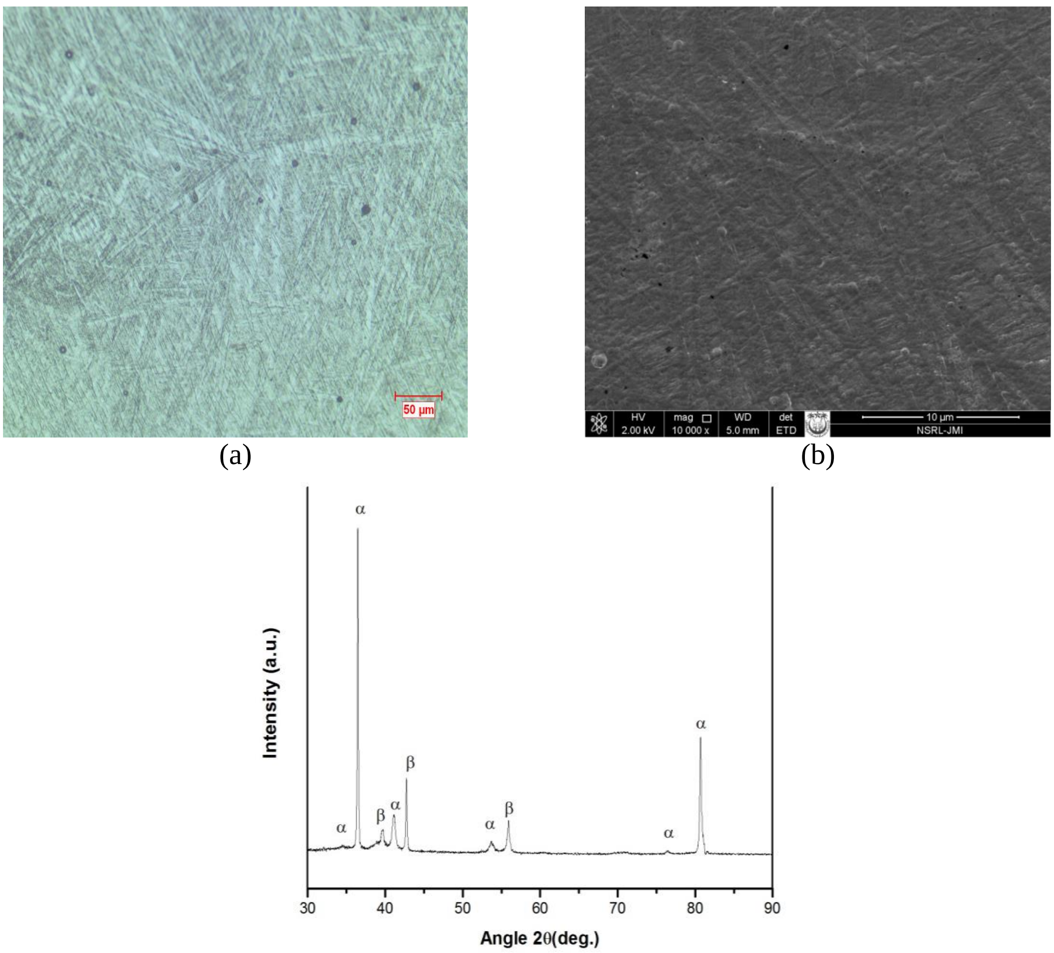
These phase constitutions were identified from XRD spectra as shown in Fig. 2(c). XRD profiles revealed peaks corresponding to only α and β phases in as-cast TNZV alloy.

4.2 Statistical analysis of experimental results

The experimental results shown in Table 3 were analyzed through *Design-Expert 6.5.0* software. Analysis of variance (ANOVA) was employed to find the effects of TMP parameters on the response variables. The details of the analysis are given in the following sections.

4.2.1 Analysis of micro-hardness

In general, high mechanical properties are supposed to be contradictory aspects for specific solid materials, particularly for metals and alloys in biomedical applications. The high hardness is necessary for the implants to meet the complex stresses including tension, compression, bending and torsion which are applied to the implants during the routine activities. The results of analysis of variance (ANOVA) for HV of TNZV alloy after TMP are shown in Table 4 which shows the sum of squares (SS), degrees of freedom (DF), mean squares (MS), F-values (F-VAL.) and "Prob>F" values. It may be noted that values of "Prob>F" less than 0.0500 indicate that the main factor and the interaction between factors significantly affect the response variable. It can be seen from Table 4 that the model is significant. Further, Table 4 also shows that within the range investigated, the working temperature (A), solution treatment temperature (B), cooling type (C), interaction (AC), interactions AB, and interaction BC have significant effect on the micro-hardness.



(a) (b)

(c)

Fig. 2 Microstructure of as-cast TNZV alloy: (a) Optical Micrograph, (b) Scanning Electron Microstructure (SEM), (c) X-ray diffraction profiles

In addition, various diagnostic tests such as normality, outlier, and residual distribution tests were performed to check the validity of the assumptions made for ANOVA.

The microstructure of the investigated TNZV alloy depends essentially upon both the hot working process and the subsequent heat treatment sequences. In the present study, thermo-mechanical processing was carried out by performing 10% forging plus 25% rolling at above β transus (850℃) and below it (650℃). The hot working at 850℃ is considered to be an effective process which produces dynamical recrystallization (DRX) with equiaxed structure (Majumdar *et al.* 2011), while hot working at 650℃ with heavy working (>30%) creates structure consisting of elongated grains in the direction of working (Freese *et al.* 2001) with simultaneous nucleation of α phase during the plastic working (Majumdar *et al.* 2011). It is expected that hot working in the $\alpha + \beta$ phase field produces low angle tilt boundary which serves as nucleation sites of α phase during subsequent cooling. The type of cooling from β transus temperature to ambient temperature establishes the morphology of phases formed which includes their volume fraction, size and shape in the microstructure. It is well known that if the Ti-alloys are cooled very slowly (furnace cooling) the adjustment of volume fraction of α phase occurs by migration of the existing α/β interface. Relatively fast cooling (air cooling) results in the formation of transformed β type structure. A non-equilibrium metastable phase called martensite can be created in Ti alloys if the cooling rate from the β phase field is sufficiently high (Banerjee and Krishnan 1981, Boyer *et al.* 1994, Majumdar 2012). Considerable literature is available (Boyer *et al.* 1994, Majumdar 2012, Collings 1984, Ahmed and Rack 1996, Hao *et al.* 2002, Mantani and Tajima 2006, Banumathy *et al.* 2011, Lee *et al.* 2013) which reports the presence of martensitic structure in Ti materials if the solution treatment has been done at high temperature (above β phase field) with sufficiently high cooling rate. It is expected that the formation of soft β and α'' martensite phases in the microstructure of water quenching plays an important role in producing lower hardness. It is pointed out (Hao *et al.* 2002) that the martensitic structure has lower hardness which is in good agreement with the present hardness result of water quenched samples. Fig. 3 shows the presence of martensitic phase in samples which were water quenched from above β transus temperature (850℃). On other hand, any increase in the volume fraction of α phase leads to significant increase in hardness. In light of the discussion given above, the present investigation has also revealed that micro-hardness (i) increases significantly with increase in the working temperature, (ii) decreases significantly with increase in the solution treatment temperature, and (iii) is maximum in case of air cooling and minimum in case of water quenching.

Table 4 ANOVA results of micro-hardness

Source	Sum of squares	DF	Mean square	F value	Prob>F
Model	4.10E+003	9	455.	455.	0.00219
A	*520.*	*1*	*520.*	*520.*	*0.00192*
B	*1.90E+003*	*1*	*1.90E+003*	*1.90E+003*	*0.000526*
C	*1.15E+003*	*2*	*576.*	*576.*	*0.00173*
AB	*397.*	*1*	*397.*	*397.*	*0.00251*
AC	*88.7*	*2*	*44.3*	*44.3*	*0.0221*
BC	*40.7*	*2*	*20.3*	*20.3*	*0.0469*
Residual	2.00	2	1.00		
Cor Total	4.10E+003	11			

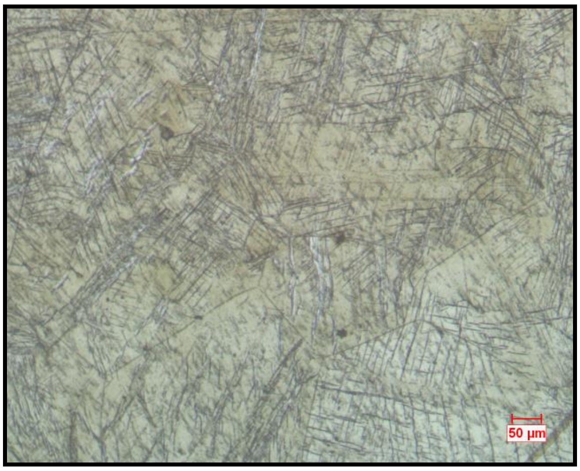


Fig. 3 Optical microstructures of the TNZV alloy deformed at 850℃ and solution treated at 850℃ for 1h followed by water quenching

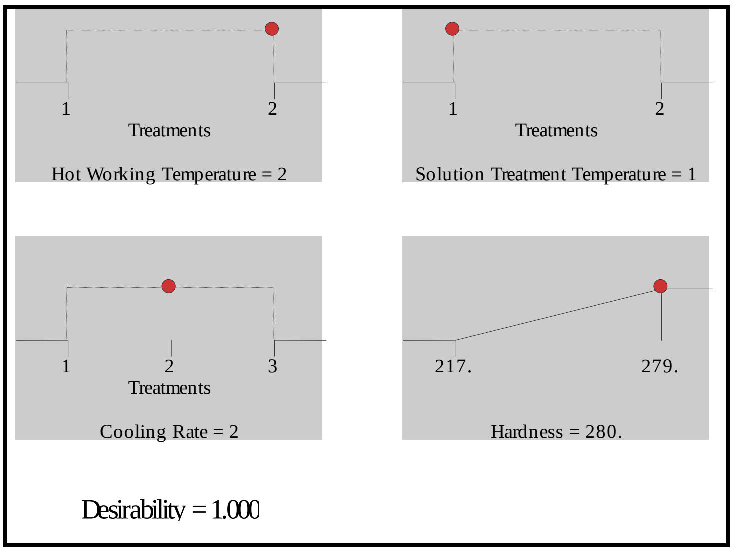
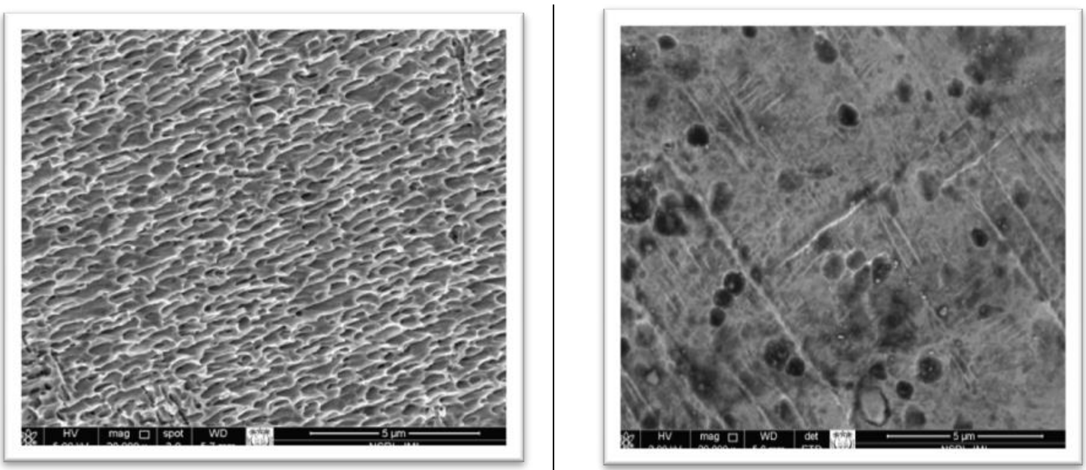


Fig. 4 Ramp function graph for micro-hardness after Thermo-mechanical processing

4.2.2 Optimization for micro-hardness

The objective of the optimization is to find the TMP parameters within the working temperature range from 650℃ to 850℃; the solution treatment temperature range from 650℃ to 850℃, and a type of cooling range from FC to WQ. TMP parameters should be such that they give maximum value of the micro-hardness. Fig. 4 shows the optimum value of TMP parameters that yields maximum value of the micro- hardness.

It can be seen from Fig. 4 that the working temperature, solution treatment temperature, and cooling type are 850℃, 650℃, and air cooling respectively which results in the optimum value of the micro-hardness (HV=279). The hardness of metals is strongly correlated to its microstructure and the microstructure developed via thermal and TMP will highly influence the



(a) (b)

Fig. 5 Scanning electron microstructures (20000X) of the TNZV alloy (a) deformed at 850℃ and solution treated at 650℃ for 1h followed by AC, (b) deformed and solution treated at 850℃ for 1h followed by WQ

micro-hardness. Amongst all heat treatment conditions, the samples which are solution treated in the (α+β) field (at 650℃) after hot working at 850℃ followed by air cooling established higher hardness owing to relatively fast cooling which formed finest distribution of α phase in β-matrix. It is well known that a small grain size of metals and alloys is usually essential to optimize the mechanical properties through controlling the microstructure and processing. The main driving force for grain growth is the reduction of the free energy coupled with the decrease in the grain boundary area (Gil and Planel 2000). The improvement in micro-hardness is achieved due to (i) grain refinement and (ii) evolution of micro-constituents (α phase). In line with findings of other researchers (Lu *et al.* 2013), the outcomes of the present research also reveals that the air cooling results in grain refinement mainly due to the Hall-Petch mechanism and dislocation density variation. The air cooling being faster than FC (but slower than WQ) resulted in relatively grain refinement as well as formation of transformed β phase both. The transformed β phase was the major contributor to the hardness. The samples which are solution treated at 850℃ (β field) after hot working at 650℃ as well as at 850℃ after hot working 850℃ followed by water quenching showed the lowest micro-hardness compared with other heat treated samples. It is reported (Hao *et al.* 2002) that the presence of martensite and β phases in the microstructure leads to decrease the hardness significantly. Consequently, it is expected that the presence of martensite with insufficient amount of α phase in the microstructure (Fig. 3 and Fig. 5(b)) led to decrease in hardness after water quenching from 850℃. The microstructure of the optimized value of micro-hardness contrasts with the experiment condition (Experimental runs 1 and 11 shown in Table 3) in which worst hardness values were obtained. The micro-graphs of both clearly reveal that in case of optimum micro-hardness the transformed β phase is significant whereas in case of worst micro-hardness it is least. The micrograph for both the cases is shown in Fig. 5.

4.2.3 Analysis of Young's modulus (E)

The results of analysis of variance (ANOVA) for Young's modulus (E) of TNZV alloy samples after TMP are shown in Table 5 which shows the sum of squares (SS), degrees of freedom (DF), mean squares (MS), F-values (F-VAL.) and “Prob>F” values. It may be noted that values of “Prob>F” less than 0.0500 indicate that the main factor and the interaction between factors significantly affect the response variable. It can be seen from Table 5 that the model is significant. Further, Table 5 also shows that within the range investigated, the solution treatment temperature (B), cooling type (C), and one way interactions (BC) have significant effect on the micro-hardness. However, working temperature (A) and two way interactions (AB) and (AC) do not significantly affect the Young’s modulus. Further, various assumptions related to the ANOVA were checked and validated through several diagnostic tests such as normality, outlier, and residual distribution tests.

The Young’s modulus is determined by the bonding force among atoms and greatly affected by the phase/crystal structure (Ho *et al.* 1999) and chemical composition of Ti material (Collings 1984, Song *et al.* 1999). Therefore, the specific modulus of the phases and by their volume fractions in any multiphase Ti alloy is a key parameter to develop the modulus. It is well known that the composition of the constituent phases and their volume fractions depend mainly upon the history of thermal and thermo-mechanical processing (Lee *et al.* 1990). It is inferred that the microstructure of Ti alloys plays a significant role in reducing the modulus as the β and α'' phase mixture lower the Young's modulus whereas the α phase increases it (Collings 1984, Cui *et al.* 2009). It is reported (Ho *et al.* 1999) that the Young's modulus of α'' phase in cast binary Ti-Mo alloys (with Mo contents close to 7.5%) has the lowest modulus among α, β, and α' phases. As discussed in the analysis of micro-hardness, Young’s modulus is also affected by solution treatment temperature, type of cooling and the interaction between solution treatment temperature and type of cooling (BC). It is reported that partitioning of alloying elements occurs during solution treatment at below transus temperature along with β to α transformation (Majumdar *et al.* 2011, Boyer *et al.* 1994, Tang *et al.* 2000). The distribution of the alloying elements depends upon their solubility, diffusion rate, as well as the time allowed for diffusion to take place (Ahmed *et al.* 2012). Therefore, β phase becomes enriched with Nb, Zr and V elements during this treatment which reduces the martensite start temperature (Ms) of the untransformed β to below room temperature (Tang *et al.* 2000) and thus no martensite was created on water quenching from 650℃. The microstructure with suppressed martinsite formation with high amount of α phase results in significant increase in the Young's modulus. However, it can be seen that the modulus of elasticity reduced significantly with an increase in cooling rate from the solution treatment temperature. After solutionizing (at 850℃) when the samples were water quenched the process

Table 5 ANOVA results of Young’s modulus

Source	Sum of squares	DF	Mean square	F value	Prob > F
Model	523.	9	58.1	36.7	0.0268
A	*0.0833*	*1*	*0.0833*	*0.0526*	*0.840*
B	*90.8*	*1*	*90.8*	*57.3*	*0.0170*
C	*284.*	*2*	*142.*	*89.5*	*0.0110*
AB	*10.1*	*1*	*10.1*	*6.37*	*0.128*
AC	*3.17*	*2*	*1.58*	*1.00*	*0.500*
BC	*135.*	*2*	*67.7*	*42.8*	*0.0228*
Residual	3.17	2	1.58		
Cor Total	526.	11			

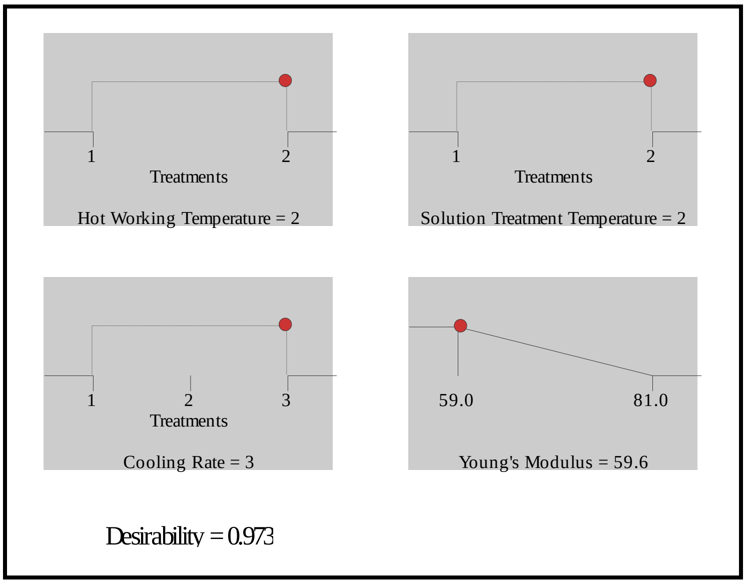


Fig. 6 Ramp function graph for Young's modulus after thermo-mechanical processing

produced a microstructure consisting of martensite and retained β phase (Fig. 3 and Fig. 5(b)). Since, both these phases offer the lowest Young's modulus as compared with α phase, the water quenched samples exhibited the lowest modulus value (59GPa) compared with furnace cooled and air cooled samples. In case of the present study, it was found that Young's modulus (i) does not decrease significantly with increase in the working temperature, (ii) decreases significantly with increase in the solution treatment temperature, and (iii) is maximum in case of furnace cooling and minimum in case of water quenching.

4.2.4 Optimization for Young's modulus

The objective of the optimization is to find the TMP parameters within the working temperature range of 650℃ to 850℃; the solution treatment temperature range of 650℃ to 850℃, and a type of cooling range from FC to WQ. TMP parameters should be such that they give minimum value of the Young's modulus. Fig. 6 shows the optimum value for TMP parameters to obtain minimum value of the Young's modulus.

It can be seen from Fig. 6 that the working temperature, solution treatment temperature, and cooling type are 850℃, 850℃, and water quenching respectively results in the optimum value of the Young's modulus (59GPa). As early discussed, solution treatment at 850℃ (β field) followed by water quenching showed the lowest value of Young's modulus compared with other heat treated samples owing to formation of soft phases of martensite and β in the microstructure which led to decrease Young's modulus significantly as evident from the microstructure shown in Fig. 3 and Fig. 5(b).

5. Conclusions

The strive for the search of compatible biomaterials is the need of the day. Development of new biomaterials, understanding their behavior during typical thermo-mechanical treatment is an effective methodology to address to this issue. In line with the urge of the day novel Ti based bio-material was developed and a comprehensive study on its thermo-mechanical treatment was performed and the effect of thermo-mechanical processing on microstructure, micro-hardness and Young's modulus of metastable-β Ti-20.6Zr-13.6Nb-0.5V alloy for biomedical applications has been statically investigated. Based on the analysis of the results, following conclusions are drawn:

•The microstructure of the thermo-mechanical treated TNZV alloy consisted mainly of equiaxed/elongated α, β phases with different morphologies and metastable martensite phase depending upon the heat treatment conditions.

•Martensitic transformations played a significant role on the reduction of the hardness and Young’s modulus.

•The optimum levels of micro-hardness are 850℃, 650℃ and air cooling for working temperature, solution treatment temperature and type of cooling.

•The optimum levels of Young’s modulus are 850℃, 850℃ and water quenching for working temperature, solution treatment temperature and type of cooling.

•According to the presented results it could be considered that the micro-hardness and Young’s modulus properties of investigated TNZV alloy suggest that the alloy is appropriate for biomedical applications.

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