Quenching temperature influence on elastic and hardness behavior in a biocompatible Ti-based alloy

Anatoliy Illarionov[1,*], *Maksim* Karabanalov[1], *Andrey* Korelin[1], and *Stepan* Novokreschenov[1]

[1]Ural Federal University, 19 Mira St., 620002 Ekaterinburg, Russia

Abstract. Elasticity modulus and hardness changes in deformed semifinished rods manufactured of biocompatible Ti-6Al-7Nb alloy under quenching from the temperatures in a range of 500-1050°C have been studied by means of indentation. The elasticity modulus and Vickers hardness have been shown to vary from 95 to 115 GPa and from 330 to 520 HV respectively depending on the quenching temperature. Relationship between the elastic and hardness behavior and the alloy phase composition has been found.

1 Introduction

The Ti-6Al-7Nb alloy [1] was purposely developed as a biocompatible material for implants to replace the Ti-6Al-4VEli alloy being widely used in medical practice. The main β-stabilizing element in Ti-6Al-7Nb is niobium because it is more biologically inert and exhibits better nontoxicity compared to vanadium [2]. Nowadays the Ti-6Al-7Nb alloy for medical implants is manufactured by means of traditional metallurgy methods, e.g., severe plastic deformation [3, 4], powder metallurgy processes [5, 6], and selective laser melting [7,8], with processing parameters greatly influencing the structure and mechanical properties of semifinished material. It should be noted that the Ti-6Al-7Nb alloy in its equilibrium state possesses rather high elasticity modulus of 105 – 110 GPa [9, 10] compared to most other Ti-based medical alloys [11, 12] because of considerable aluminum content, and this somewhat reduces the alloy biocompatibility for implant application. Quenching from different temperatures corresponding to two-phase (α + β)-field was shown by using the VT16 Ti-based medical alloy [13] to result in considerable elasticity modulus variation due to obtaining various phase composition. As for the Ti-6Al-7Nb alloy, its phase composition also changes with the quenching temperature corresponding to two-phase (α + β)-field [14], but the influence of those changes on the alloy elasticity modulus and hardness has not been studied yet. It is within the context of the above that the aim of this research is to study the influence of quenching temperature on the deformed semifinished Ti-6Al-7Nb alloy elasticity modulus and hardness, with the quenching being carried out from two-phase (α + β)- and single-phase β-fields, and to consider the relationship between the elastic and hardness behavior and the phase composition of the alloy quenched.

2 Materials and Methods

Hot-rolled rods (20 mm in diameter) supplied by VSMPO-AVISMA Corporation were manufactured of the Ti-6Al-7Nb alloy with the composition of Ti-6,28Al-7,24Nb-0,19O–0,19Fe-0,015C-0,009N-0,001H (wt.%) [14] and subjected to the deformation in two-phase (α + β)-field.

The temperature of complete polymorphous α + β → β transformation (T_{pt}) determined by means of test quenching was 990±5 °C. The heat treatment procedure of the Ti-6Al-7Nb alloy specimens consisted of heating up to the temperatures in a range of 500-1050°C with the interval of 50°C, holding during 1 hour, and water quenching.

The elasticity modulus and hardness were measured by means of indentation using MHTX equipment from CSM instruments at a load of 9N, with calculations made according to Oliver-Pharr technique [15]. X-ray diffraction analysis was carried out using a DRON 3M X-ray diffractometer and a Neophot 2 light microscope.

3 Results and Discussion

The Ti-6Al-7Nb alloy structure analysis in the initial hot rolled alloy state has revealed that the structure mostly consists of the bright primary α-phase precipitates of rather complex shape which is sometimes similar to the globular one (Fig. 1). Space around the primary α-phase particles is occupied by the matrix turned into β-phase, with the increased matrix etchability making it darker compared to the primary α-phase. There are the second

* Corresponding author: illarionovag@mail.ru

© The Authors, published by EDP Sciences. This is an open access article distributed under the terms of the Creative Commons Attribution License 4.0 (http://creativecommons.org/licenses/by/4.0/).

α-phase precipitates (dispersed particles) within the above matrix (Fig. 1).

It has been found by means of X-ray diffraction analysis that the alloy structure in its hot rolled state includes only α- and β-phases with the following parameters: a_α=0.29298 nm, c_α=0.46748 nm, c/a=1.5956, a_β=0.32577 nm. The c/a parameter obtained for the α-phase is higher than that of pure titanium (1.587 [16]), with this fact being characteristic for the aluminium doped alloys [17]. The β-phase lattice constant is rather high, and this can be observed when the high-temperature β-solid solution does not break down completely during its cooling from its last treatment temperature. The values of elasticity modulus (105 GPa) and hardness (345 HV) in the hot rolled state are typical for the alloy [10, 18].

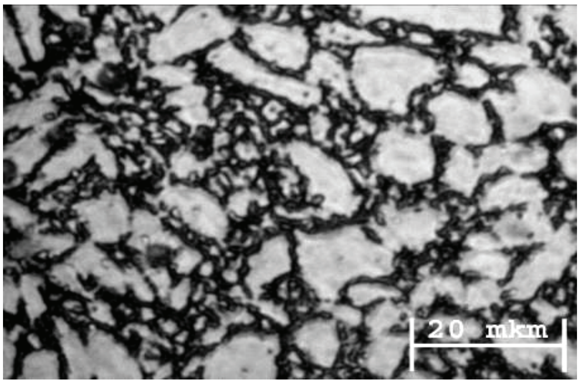

Fig. 1. Structure of as-hot rolled Ti-6Al-7Nb alloy

Figure 2 shows the quenching temperature dependences of the Ti-6Al-7Nb alloy hardness and elasticity modulus measured by means of indentation. These temperature dependences reveal their minimum after the quenching from 750°C. The alloy hardness and elasticity modulus decrease from 430 to 330 HV and from 105 to 95 GPa respectively with increasing quenching temperature from 500 to 750°C, whereas the alloy hardness and elasticity modulus grow up to 500-520 HV and 115 GPa respectively at the higher quenching temperatures in a range of 800-1000°C. The alloy hardness values obtained after quenching are always higher (Fig. 2a), whereas the alloy elasticity modulus values are higher only after quenching from the temperatures in a range of 950-1050°C (Fig. 2b) compared to the above values in the initial hot-deformed alloy state.

The quenching temperature dependences of the Ti-6Al-7Nb alloy hardness and elasticity modulus (Fig. 2) are consistent with the changes of the alloy phase composition (Table 1, [14]) and the lattice constant of the metastable phases observed under quenching (Table 2, Fig. 3).

Table 1. Ti-6Al-7Nb alloy phase composition after quenching from different temperatures [14]

Quenching temperature, °C	500-700	750	800-900	950	1000-1050
Phase composition	α+β	α+α''+ β	α+α''	α+α'	α'

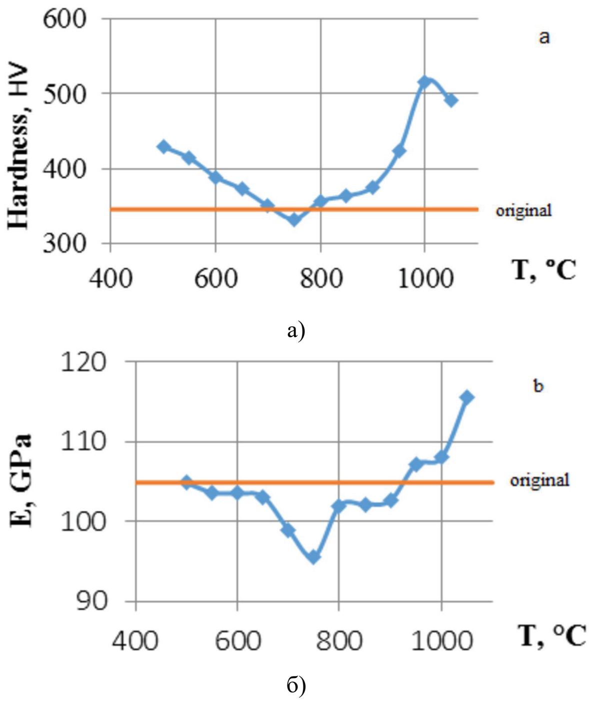

Fig. 2. Change of (a) hardness and (b) elasticity modulus in Ti-6Al-7Nb alloy with the quenching temperature.

Table 2. β-phase lattice constant after quenching

Tq,°C	500	550	600	650	700
a_β, nm	0.32435	0.32414	0.32465	0.32477	0.32508

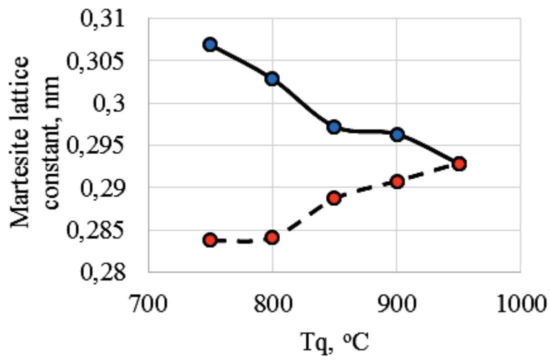

Fig. 3. Change of martensite lattice constant in Ti-6Al-7Nb alloy with the quenching temperature ($a_{\alpha''}$ – solid line, (b/v3)$_{\alpha''}$ – dashed line).

The Ti-6Al-7Nb alloy structure includes α- and β-phases under the quenching at the temperatures in a range of 500-700 °C, with the β-phase lattice constant in the temperature range mentioned above being lower than that in the initial hot-deformed alloy state. This β-phase lattice constant reduction is obviously connected with the breakdown in the initial β-solid solution during the holding in the quenching temperature range mentioned above. The breakdown is accompanied by the second α-phase precipitation which contributes to obtaining higher alloy hardness values compared to those in the initial alloy state. With increasing heating temperature before the quenching in the temperature range mentioned above the second phase precipitates are coarsening and their volume fraction is decreasing (the fact is proved by the β-phase lattice constant growth), the hardening effect is reducing reaching its minimum at the temperature of 700°C. At the quenching temperature of 750°C the metastable β-phase solid solution being observed during the heating before quenching does not break down but partly undergoes the martensitic β → α''-transformation during the cooling. In this case the fact that the second α-phase precipitations do not exist and the α''-martensite which is rich enough in alloying elements but is known [17] not to possess high hardness is observed leads to obtaining minimal hardness values over the whole quenching temperature range studied. The further quenching temperature increase up to 900°C results in the complete martensitic β → α''-transformation of the β-phase solid solution during quenching, but according to [14] the forming α''-martensite is less rich in β-stabilizing elements (niobium, iron), exhibits less rhomboidity (being characterized by the difference in $a_{\alpha''}$ and $(b/\sqrt{3})_{\alpha''}$ values, see Fig.3) but higher hardness. This contributes to some growth of the alloy hardness values compared to those in its hot rolled state. At the quenching temperature of 950°C and higher the β-phase solid solution undergoes the martensitic β → α'-transformation. The α'-martensite is shown in [17] to possess the best hardness behavior compared to β-, α''-, and α-phases, it is this fact which leads to the considerable hardness growth compared to the alloy hardness in its hot rolled state with increasing quenching temperature up to T_{pt}. When quenching from the temperatures higher than T_{pt} the alloy hardness somewhat decreases because the β-grain size was shown by microstructure to grow from 600 μm at T_q = 1000°C to 900 μm at T_q = 1050°C, and the forming α'-martensite needles coarsen correspondingly. The similar hardness dependence on quenching temperature along with the martensite formation was observed in the VT18U alloy [19] which contains about 6% of Al as well as the Ti-6Al-7Nb alloy does but less amount of Nb (1.13%).

Unlike hardness the Ti-6Al-7Nb alloy elasticity modulus value after the quenching from 500°C (105 GPa) coincides with that of the hot rolled semifinished material. This is due to fact that the elasticity modulus in not influenced by the alloy structural features (e.g., existence of the second phase precipitates) [20], but it is connected with the alloy phase composition which remains the same after the quenching from 500°C and in the hot rolled alloy state. Meanwhile, the alloy elasticity modulus value tends to reduce with increasing quenching temperature up to 750°C which is due to the β-phase solid solution volume fraction growth observed in [14] under quenching. (Note that the β-phase solid solution exhibits low elasticity modulus.) The further quenching temperature increase up to 1050°C results in the fact that the alloy elasticity modulus value tends, on the contrary, to grow (Fig.2b). This is connected with the following: the α''-martensite forming under the quenching from the temperatures lower than 950°C is becoming less alloyed by β-stabilizing elements (as shown above, Fig.3) with increasing quenching temperature but, at the same time, the α''-martensite elasticity modulus was established in the VT16 alloy [13] to be growing. However, it should be noted that the alloy elasticity modulus value is less than that in the initial hot rolled alloy state because the elasticity modulus of the α-phase being the main part of the initial hot rolled alloy structure is higher than that of the α''-martensite forming under the quenching. After the quenching from the temperature of 950°C and higher the α'-martensite is observed in the alloy structure (Table 1), it exhibits higher elasticity modulus [19] compared to that of α-phase and, therefore, the alloy elasticity modulus values obtained are higher than those in the initial hot rolled alloy state and increase up to T_{pt} due to reducing primary α-phase volume fraction and growing α'-martensite volume fraction [14].

On the whole, the Ti-6Al-7Nb alloy elasticity modulus values vary from 95 to 115 GPa over the quenching temperature range studied. The minimal value of 95 GPa is 10-15 GPa less than that of the alloy in its equilibrium state and obtained after the quenching from 750°C which is close to this alloy critical temperature (T_c), the quenching from any temperature above T_c leads to the β-phase solid solution martensitic transformation [21]. The maximum elasticity modulus value of 115 GPa is 5-10 GPa higher than that of the alloy in its equilibrium state and obtained after the quenching from above T_{pt}, from β-phase field with T_q = 1050°C, with the 100% α'-martensite structure being observed. It follows from the above that to obtain the Ti-6Al-7Nb alloy elasticity modulus values about 95 GPa it is necessary to use heat treatment including the quenching from the temperatures close to T_c.

4 Conclusions

Thus, the following conclusions can be made based on the research carried out:

1. The as-hot rolled Ti-6Al-7Nb alloy is a two-phase α + β-alloy consisting predominantly of the primary α-phase of complex, near to globular morphology which is imbedded into the matrix turned into β-phase with the second α-phase precipitates. The alloy possesses its typical values of hardness (345 HV) and elasticity modulus (105 GPa).
2. The temperature dependences of the alloy hardness and elasticity modulus within the temperature range of 500-1050 °C have been found to show their minimum at 750 °C which is close to this alloy critical temperature. The ranges of the alloy Vickers hardness and elasticity modulus variations under quenching are 330-520 HV and 95-115 GPa respectively.
3. It has been shown that the alloy hardness variation under quenching depends on both its structural (whether or not the second phase precipitates exist) and phase transformations, whereas the alloy elasticity modulus variation is mostly influenced by its phase composition changes.

We hereby acknowledge the support of the Ministry of Science and Education of the Russian Federation in accordance to the decree of the Government of April 9, 2010, №218, project number 02.G25.31.0234.

References

1. M. Semlitsch, F. Staub, H. Weber, Biomed. Technik, (30), 334-339 (1985)
2. A. Biesiekierski, J. Wang, Mohamed Abdel-Hady Gepreel, C. Wena. Acta Biomater, 8, 1661 (2012)
3. P. Barbosa, S. Button, Proceedings of the Institution of Mechanical Engineers Part L: Journal of Materials: Design and Applications, 214, 23-31, (2000)
4. V. Polyakova, I. Semenova, R. Valiev, Materials Science Forum, **667-669**, 943-948 (2011)
5. V. Henriques, C. Da Silva, Key Engineering Materials, **189-191**, 443-448 (2001)
6. L. Bolzoni, E. Ruiz-Navas, E. Gordo, Journal of the Mechanical Behavior of Biomedical Materials, 67, 110-116 (2017)
7. T. Sercombe, N. Jones, R. Day, A. Kop, Rapid Prototyping Journal, 14, 300-304 (2008)
8. E. Chlebus, B. Kuźnicka, T. Kurzynowski, B. Dybała, Materials Characterization, 62, 488-495 (2011)
9. M. Niinomi, Materials Science and Engineering A, 243, 231-236 (1998)
10. R. Boyer, G. Welsch and E. Collings *Materials Properties Handbook*: *Titanium Alloys*. Ed. by, *ASM International*, (Materials Park, OH, USA, 1994)
11. M. Geetha, A. Singh, R. Asokamani, A. Gogia, Progress in Materials Science, 54, 397-425 (2009)
12. C.-W. Lin, C.-P. Ju, J.-H. Lin, Materials Transactions, 45, 3028-3032 (2004)
13. A. Popov, A. Illarionov, S. Stepanov, O. Ivasishin, Physics of Metals and Metallography, 115, 517-522 (2014)
14. A. Illarionov, M. Karabanalov, S. Stepanov, Metal Science and Heat Treatment, 52, 481-486 (2011)
15. W. Oliver, G. Pharr, Journal Materials Research, 7, 1564-1583 (1992)
16. U. Zwicker, *Titanium and its alloys* (Metallurgija, Moscow, 1979)
17. A. Popov, A. Illarionov, N. Rossina, S. Grib, Metal science and heat treatment of titanium alloys. Structure and properties, UrFU (2012)
18. D. Gadeev, A. Illarionov, S. Demakov, Metal Science and Heat Treatment, 57, 463-468 (2015)
19. L. Bolzoni, E. Ruiz-Navas, E. Neubauer, E. Gordo, Journal of the Mechanical Behavior of Biomedical Materials, 9, 91-99 (2012)
20. B. Livshits, Physical properties of metals and alloys, Textbook, 2nd ed., Ext. and rev. (1980)
21. N.F. Anoshkin, G.A. Bochvar and others, *Metallography of titanium alloys* (Metallurgia, Moscow, 1980)