DETERMINATION OF A SUITABLE HEAT TREATMENT METHOD FOR THE TITANIUM ALLOY Ti-6Al-4V ENDOPROSTHESIS SHANK

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Abstract

The article concerns the experimental determination of a suitable method of heat treatment of the shank of a hip arthroplasty, that is, an artificial replacement of the femoral head with the neck, to obtain the mechanical properties required by the customer. The hip replacement shank is manufactured from Ti-6Al-4V titanium alloy. As the customer's requirements for the mechanical properties of the hip replacement shank were not met after the hardening process, in this case quenching in water and subsequent tempering, repeated annealing was experimented in a Realistic 2 continuous oven. After each alternative annealing of the hip replacement shank, three circular cross-sectional test bars were made, and then the mechanical properties of the material were evaluated by tensile testing. The tests carried out show that after the first annealing, the mechanical properties of the material and its microstructure may not be satisfactory and, therefore, it is advisable to repeat the annealing. In the present case of a suitable heat treatment method for the shank of a hip arthroplasty, the customer's requirements for its mechanical properties and its microstructure after the third annealing were met.

Keywords: Heat treatment, titanium alloy, endoprosthesis shank, annealing

1. INTRODUCTION

Titanium and its alloys are used only in certain industries where other more affordable sources cannot be used. The most widely used titanium alloy is Ti-6Al-4V because of its high strength, low specific gravity, creep resistance, crack propagation resistance or corrosion resistance. These properties can be significantly affected by heat treatment [1–4]. Titanium alloys are heat treated to achieve the following: Stress relief to reduce residual stresses developed during fabrication. Annealing to achieve an optimum combination of ductility, machinability, dimensional stability, and structural stability. Treatment and ageing of solution, to increase strength.

The Ti-6Al-4V alloy is used in aerospace, marine and energy industries and, due to its biocompatibility, also in medicine [5]. The titanium alloy Ti-6Al-4V is used for the production of hip arthroplasty shanks by hot volume forming followed by heat treatment. The forgings have good mechanical properties, unbroken fibres that follow the complexity of the shape of the component, and there is little waste during production. The disadvantage is the energy consumption and high cost of producing single-purpose tooling, namely dies. The useful life of forging tools is affected by their resistance to thermal and abrasive wear [6,7]. The prevention of defects in forging tools is possible by using non-destructive detection methods [8]. A suitable forging machine should be selected according to the size of the batch and the required properties [9]. Crank presses are more powerful than hammer presses; they also do not need such massive foundations and have less demands on the qualification and physical strength of the operator. Blasting or tumbling [10] can be used to remove forging burrs and scale. In the manufacturing process, a properly designed maintenance organisation [11–13] and the monitoring and prevention of defects [14] are important. The achievement of maximum production productivity can be achieved, for example, by a good design of the distribution warehouse [15].

2. TITANIUM ALLOY ENDOPROSTHESIS SHANKS Ti-6Al-4V

Hip endoprostheses shanks (**Figure 1**), which are part of hip replacement (**Figure 2**), are manufactured in the joint stock company Alper Forge by forging with subsequent heat treatment. A hip replacement consists of a socket (replacing the original socket in the pelvis), a shank (located in the femur), a head, and an articulating insert (most commonly modified polyethylene and ceramic). Some implants are fixed in bone using bone cement, others without. As a rule, cemented endoprostheses are implanted in older patients and uncemented in younger patients.

Figure 1 Hip arthroplasties shafts made of Ti-6Al-4V alloy (photo by Radek Čada)

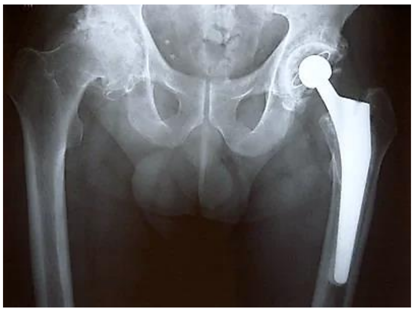

Figure 2 X-ray of an implanted hip replacement (source: cs.medlicker.com)

The inert material used for hip arthroplasty shanks is the Ti-6Al-4V titanium alloy according to ISO 5382-3 (see **Table 1**), also known as Ti64. Ti-6Al-4V, classified as a two-phase α+β alloy, is the most widely used titanium alloy. At normal temperature, their microstructure is composed of both α and β phases. The additive elements most commonly used in these alloys are aluminium (α stabiliser) and vanadium (β stabiliser). Aluminium dissolves hydrogen well, thus suppressing hydrogen corrosion. With a higher aluminium content, embrittlement increases. Vanadium dissolves well in the β phase and increases strength and ductility in titanium alloys. Some mechanical properties may be affected by heat treatment.

Table 1 Requirements of the ISO 5382-3 standard for the mechanical properties and microstructure of the wrought titanium alloy Ti-6Al-4V (in bars) for use in the manufacture of surgical implants

$R_{p0,2}$ (MPa)	R_m (MPa)	A_5 (%)	**Microstructure (α + β globular)**
min. 780	min. 860	min. 10	A1 to A9 according to ISO 20160

The Ti-6Al-4V alloy provides a combination of high strength, low weight, ductility, and corrosion resistance. This alloy also exhibits good mechanical properties at low temperatures and very good resistance to crack initiation and propagation (resistance to material fatigue). Due to the protective layer of oxidation on the surface, Ti-6Al-4V is resistant to corrosion even in aggressive environments. A positive feature is its biocompatibility with human tissue. The Ti-6Al-4V alloy is suitable for forming heat transfer in the α+β region in the temperature range 870–980 °C. When heat transfer is formed in an atmosphere containing nitrogen or oxygen, these elements diffuse into the surface layer of the material, causing it to harden. This hard and brittle layer is removed after forming by machining or picking.

3. TECHNOLOGY OF MANUFACTURING OF SHANKS OF HIP JOINT ENDOPROSTHESES

The technology to produce hip joint endoprosthesis shanks in the joint stock company Alper Forge is as follows. From the supplied metallurgical semifinished product Ø35 x 250 mm, which is heated in the inductor to the forging temperature of 1020–1070 °C, the endoprosthesis shank is forged in a vertical forging press LKZ 2500 (Smeral, Brno, Czech Republic) (**Figure 3**) in three operations:hot bending, ramming, finishing of the forging (**Figures 4 and 5**). After forging, the forging is cut from the projection (**Figure 5**). After the forging has cooled, heat treatment follows. The usual batch size is 125 pieces.

Figure 5 Vertical forging press LKZ 2500

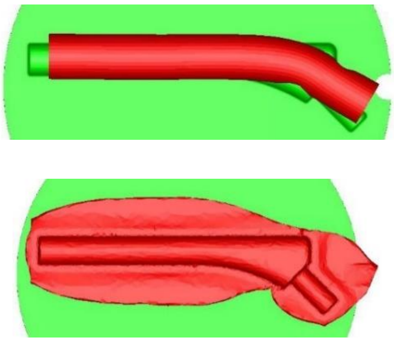

Figure 6 Filling (top) and finishing (bottom) the endoprosthesis shank forging in the die

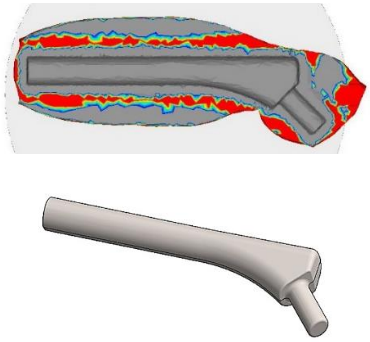

Figure 7 Contact of the forging with the die cavity at the end of the forging (top) and endoprosthesis shank model (bottom)

In the past, the jointstock company Alper Forge used a hardening temperature of 850–880 °C for the shanks of titanium alloy Ti-6Al-4V endoprostheses, from which hardening in water was carried out. The quenching was followed by tempering at 450–550 °C for 4 hours, where the lower temperature resulted in a higher hardening of the alloy. After tempering, the endoprosthesis shanks were cooled in air for 4 to 5 hours. As the above heat treatment of the endoprosthesis shanks resulted in too high strength values, subsequent annealing with recrystallisation and slow cooling in air was necessary. As the described heat treatment process was energy intensive, an annealing heat treatment process was proposed and included after the forging was completed. The final surface treatment of the endoprosthesis shanks is performed at the customer site.

4. EXPERIMENTAL DETERMINATION OF A SUITABLE METHOD OF HEAT TREATMENT OF THE SHANK OF HIP ARTHROPLASTY

The experimental determination of a suitable method of heat treatment of a shank of a hip arthroplasty, ie an artificial replacement of the femoral head with neck, made of Ti-6Al-4V titanium alloy to obtain the mechanical properties required by ISO 5382-3, was carried out in the joint stock company Alper Forge.

The chemical composition (**Table 2**) was determined and the microstructure was evaluated for the metallurgical billet Ø35 x 250 mm supplied. Metallographic analysis was performed on a transverse section in accordance with ISO 20160. The etchant used to induce the microstructure was 1 % HF + 2 % HNO_3 + 95 % glycerine. The initial state microstructure of the Ti-6Al-4V alloy delivered in the annealed state was globular, consisting of a predominantly equiaxed α phase (with a hexagonal atomic structure) and a small amount of β phase (with a body centred cubic atomic structure) of irregular shape along the grain boundaries of the α phase.

Table 2 Requirements of the ISO 5382-3 standard for the chemical composition of the wrought titanium alloy Ti-6Al-4V (in bars) for use in the manufacture of surgical and chemical composition of the starting semifinished product for shank (wt%)

Element	Al	V	Fe	O	C	N	H	Ti
Compositional limits	5.5 to 6.75	3.5 to 4.5	max. 0.3	max. 0.2	max. 0.08	max. 0.05	max. 0.015	Balance
Starting semi-finished product	6.45	4.08	0.22	0.164	0.008	0.004	0.005	Balance

As per the requirements of the company BEZNOSKA, Ltd. for the mechanical properties and microstructure of the hip replacement shank (mechanical properties according to **Table 1** and microstructures A21 to A24 according to ISO 20160 are required) were not met after hardening; in this case hardening in water and subsequent tempering, repeated annealing was tested: the first 900 °C/air, the second 900 °C/air, and the third 880 °C/air. The annealing of the endoprosthesis shank forgings was carried out in a Realistic 2 continuous furnace (**Figure 6**) without an inert atmosphere. It is a combined furnace that has a gas part and an electric part. Sequential heating was performed on the gas part of the furnace consisting of four sections. The temperatures in the four sections in succession were set at 780 °C, 880 °C, 900 °C and 900 °C with a total oven pass time of 120 minutes. After passing through the gas section of the continuous oven, the endoprosthesis forgings were continuously cooled in still air in the off-electric section of the oven.

Figure 6 Inlet of the Realistic 2 Continuous Furnace

After each alternative annealing of the hip replacement shank, three circular cross-sectional test bars were produced on a SU 50A/1000 tip lathe using a turning knife with a sintered carbide insert designed for machinability class 14b, and then the mechanical properties of the material were evaluated by tensile tests according to EN ISO 6892-1 as the arithmetic mean of the three measured values. To verify the heat treatment process of the hip arthroplasty shank, tensile tests of circular cross-section test rod with a diameter of 10 mm were performed on a UHS 60 universal test machine (Losenhausenwerk Düsseldorf, Germany) with a maximum tensile force of 600 kN with an accuracy of ± 0.2 kN at a temperature of 20 °C.

For the tensile tests, one test rod was made from a single piece of the hip arthroplasty shank (**Figures 7** and **8**). Due to the high cost of the Ti-6Al-4V alloy, it was proposed that in the future tensile tests would be performed using 6mm diameter threaded test rods, allowing two test rods to be obtained from one piece of hip shank (**Figure 8**).

The hardness after heat treatment was measured with an HPO 3000 hardness tester with a 5 mm diameter carbide ball under a load of 750 N with a loading time of 10 s according to EN ISO 6506-1.

Figure 7 Shanks of hip arthroplasty after removal of the test rod section (the border of the cylindrical test rod section is marked)

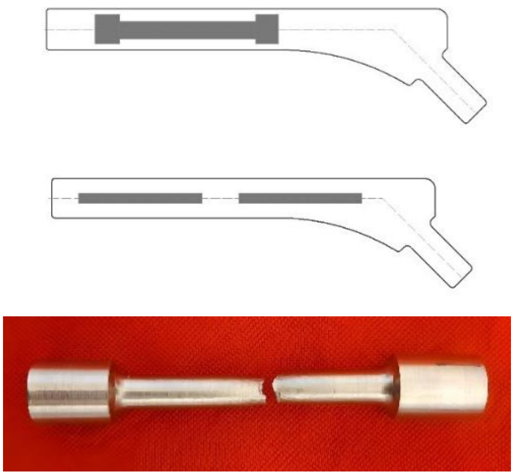

Figure 8 Position of the test rods (top 10 mm diameter, bottom 6 mm diameter) in the endoprosthesis shank and the test rod after the tensile test

Figure 9 Fracture surface on test bar after first annealing (top) and after third annealing (bottom)

5. RESULTS AND THEIR DISCUSSION

The average values of the mechanical properties after the reannealing process calculated from three measurements for each heat treatment alternative are summarised in **Table 3**. The tests carried out show that after the first annealing at 900 °C/air/120 minutes, the mechanical properties and microstructure of the material may not be compatible with customer requirements and therefore the annealing should be repeated. Based on the test results (see **Table 3**), it can be seen that the repeated annealing process does not have a significant effect on the hardness and ultimate strength of the shank material, but the yield strength and ductility values increase. In the case under consideration, the customer's requirements for the mechanical properties and microstructure of the shank material after the third annealing were met to determine the appropriate heat treatment method for the hip replacement shank (see **Table 3**).

Table 3 Values of mechanical properties of hip replacement shank forgings of Ti-6Al-4V titanium alloy after repeated annealing process

Heat treatment order	Annealing parameters	Hardness HBW	Yield stress $R_{p0,2}$ (MPa)	Tensile strength R_m (MPa)	Ductility A_5 (%)	Microstructure according to ISO 20160
1	900 °C/air/120 minutes	293 ± 1	607 ± 2	871 ± 2	12.4 ± 0.3	A17
2	900 °C/air/120 minutes	293 ± 1	716 ± 2	882 ± 2	13.1 ± 0.4	A19
3	880 °C/air/120 minutes	295 ± 1	828 ± 3	883 ± 2	17.0 ± 0.5	A22

The endoprosthesis shank is forged in a vertical LKZ 2500 forging press from a semi-finished product that is heated in an inductor to a forging temperature of 1020–1070 °C. This is higher than the phase transformation temperature (980–1011 °C). As forging is followed by cooling in air, a duplex type of microstructure is formed, consisting mainly of a transformed β phase (lamellar structure) and a small amount of globular α phase formed inside the lamellar structure. Subsequent annealing of the lamellar structure at temperatures below the phase transformation temperature leads to hardening of the β lamellae by a fine secondary lamellar α phase (see **Figure 11**). With each subsequent annealing, this process of precipitation of a new α phase continues, increasing the yield strength of the material. The microstructure of the Ti-6Al-4V alloy after the third annealing is shown in **Figure 12**. The finer microstructure of the forged shank after the first annealing (**Figure 11**) and the coarser microstructure of the forged shank after the third annealing (**Figure 12**) are consistent with the less rugged fracture surface relief on the test bar after the first annealing (**Figure 9 top**) and the more rugged fracture surface relief on the test bar after the third annealing (**Figure 9 bottom**).

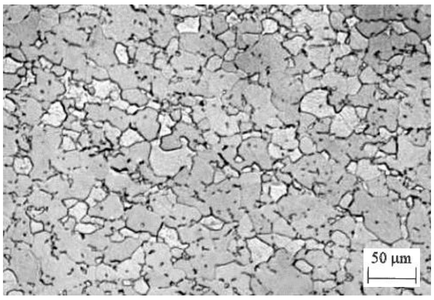

Figure 10 Microstructure of the initial metallurgical semi-finished product

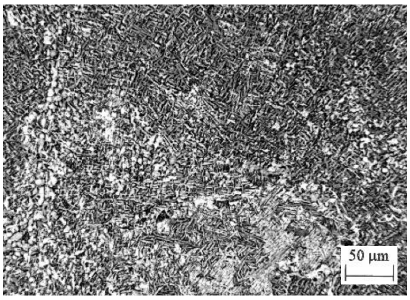

Figure 11 Microstructure of the forged shank after the first annealing

Figure 12 Microstructure of the forged shank after the third annealing

During forging in air and during annealing without a protective atmosphere, the Ti-6Al-4V alloy is contaminated with elements (O, N, C, H), which leads to hardening of the surface layer of the material. In particular, hydrogen readily diffuses into the material and causes embrittlement of its surface layers. In the case of an endoprosthesis stem, this hardened surface layer is removed by subsequent machining.

6. CONCLUSIONS

According to the results of the tests, it can be stated that forgings made of Ti-6Al-4V alloy can be heat treated in continuous furnaces without protective atmosphere by an annealing process. It has been experimentally verified that in the case of a shank forging of a hip arthroplasty made of titanium alloy Ti-6Al-4V, it is possible to increase the values of mechanical properties by precipitating a new phase α and changing the microstructure to the one required by the customer by repeated annealing under the phase transformation temperature.

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REFERENCES

[1] AHMED, T., RACK, H.J. Phase transformations during cooling in α+β titanium alloys. *Materials Science and Engineering.* 1998, vol. 243, no. 1–2, pp. 206-211.

[2] LÜTJERING, G. Influence of processing on microstructure and mechanical properties of (α+β) titanium alloys. *Materials Science and Engineering.* 1998, vol. 243, no. 1–2, pp. 32-45.

[3] OMONIYI, P.O., AKINLABI, E. T., MAHAMOOD, R. M. Heat treatments of Ti6Al4V alloys for industrial applications: An overview. In: *International Conference on Engineering for Sustainable World (ICESW 2020*). Ota, Nigeria: IOP Conf. Ser.: Mater. Sci. Eng. 2021, vol. 1107, Article 012094.

[4] FISARA, S. *Influence of heat treatment on microstructure of TiAl6V4 alloy.* Bachelor work. Brno: VUT in Brno, Faculty of Mechanical Engineering. 2016, 43 p. In Czech.

[5] CADA, R., FRYDRYSEK, K., SEJDA, F., TILLER, P. Evaluation of locking bone screw heads. In: *Proceedings of the 24th International Conference on ENGINEERING MECHANICS 2018 (May 14–17, 2018, Svratka, Czech Republic).* Editors: Fischer, C. and Náprstek, J. Prague: Institute of Theoretical and Applied Mechanics of the Czech Academy of Sciences, 2018, pp. 133-136, Paper #361.

[6] EVIN, E., TOMAS, M. Tribology properties evaluation for friction pair Zn coated steel-TiCN MP coated/uncoated tool steel. *Acta Metallurgica Slovaca*. 2019, vol. 25, no. 4, pp. 208-216.

[7] TAVODOVA, M., HNILICA, R. Assessment of selected properties of treated tool surfaces examined to increase tool life time. *Manufacturing Technology*. 2020, vol. 20, no. 2, pp. 257-264.

[8] STANCEKOVA, D. et al. Investigation of defects in forging tools by non-destructive detection method. In: 22*nd* *International Conference on Metallurgy and Materials (METAL 2013).* Ostrava, Czech Republic: TANGER, Ltd., 2013, pp. 485-490.

[9] CADA, R., MATEJ, S. Solution of a suitable method of production of contactor forgings. *Modern Machinery Science Journal.* 2023, vol. 16, no. 1, pp. 6304-6313.

[10] CADA, R., LOSAK, P. Comparison of deburring technologies after pipe cutting. In: *Proceedings of the 30th Anniversary International Conference on Metallurgy and Materials (METAL 2021).* 1. Ed. Brno: TANGER, Ltd., 2021, pp. 585-591.

[11] NECAS, L. Training and practice to ensure implementation of the TPM system. *Modern Machinery Science Journal*. 2019, vol. 12, no. 2 (June), pp. 4124-4127.

[12] NECAS, L., NOVAK, J. Networking of maintenance entities for production and other processes. *Modern Machinery Science Journal.* 2021, vol. 14, no. 6, pp. 5410-5413.

[13] SCHINDLEROVA, V., SAJDLEROVA, I. Comparison of basic maintenance concepts using witness. *Modern Machinery Science Journal*. 2021, vol. 14, no. 6, pp. 5435-5440.

[14] SPROCH, F., NEVIMA, J. Increasing competitiveness through innovation in an enterprise: A case study of the company massag. *Advances in Science and Technology-Research Journal.* 2021, vol. 15, no. 1, pp. 110-125.

[15] SCHINDLEROVA, V., SAJDLEROVA, I., BUCKO, M. Potential of using simulation for the design of distribution warehouse. In: *8th Carpathian Logistics Congress on Logistics, Distribution, Transport and Management (CLC).* Prague, Czech Republic: TANGER, Ltd., 2019, pp. 802-807.