Advances in Science and Technology Research Journal
Volume 11, Issue 4, December 2017, pages 147–158
DOI: 10.12913/22998624/76488

Research Article

INVESTIGATION OF THE EFFECT OF FORGING TEMPERATURE ON THE MICROSTRUCTURE OF GRADE 5 TITANIUM ELI

Anna Dziubińska[1], Krzysztof Majerski[2], Grzegorz Winiarski[1]

[1] Lublin University of Technology, Mechanical Engineering Faculty, Department of Computer Modelling and Metal Forming Technologies, Nadbystrzycka 36, 20-618 Lublin, Poland, e-mail: a.dziubinska@pollub.pl

[2] Lublin University of Technology, Mechanical Engineering Faculty, Department of Materials Engineering, Nadbystrzycka 36, 20-618 Lublin, Poland

Received: 2017.08.18
Accepted: 2017.11.01
Published: 2017.12.05

ABSTRACT
The paper reports the experimental results of an investigation of the effect of forging temperature on the microstructure and hardness of Grade 5 titanium ELI. A growing interest in the use of titanium alloys in implantology results from their unique properties. Grade 5 titanium ELI is widely used in medicine for producing a variety of implants and medical tools such as hip, knee and shoulder joints; bone plates; pacemaker casing and its components; screws; nails; dental materials and tools. In the first part of the paper the properties of Grade 5 titanium ELI are described and examples of medical applications of this alloy are given. In a subsequent section of the paper, forging tests performed on this biomaterial in a temperature range from 750°C to 1100°C are described. Following the forging process, the results of the titanium alloy's microstructure and hardness are reported. The experimental results are used to determine the most suitable forging temperature range for Grade 5 titanium ELI with respect to its microstructure.

Keywords: grade 5 titanium ELI, biomaterials, metal forming, forging, microstructure, hardness

INTRODUCTION

A growing interest in the use of titanium alloys as biomaterials in medicine stems from their unique properties including [3, 5, 9, 12–13, 15–16, 28, 23]:

- low density;
- high tensile strength;
- high corrosion resistance;
- optimum biocompability when compared to other metallic biomaterials;
- repassivation of surface damage in wet and oxygen-containing environment;
- higher elasticity than other biomaterials, which means that the implant-adjacent bone can continue to perform its load-carrying functions;
- promotes osseointegration;
- titanium implants can be used on a long-term basis (for 20 years at least);
- the possibility of monitoring titanium implants by resonant methods.

Given the general market availability of titanium and its alloys due to advanced metallurgical methods and machining techniques, titanium alloys are widely used in orthopaedics, traumatology and dentistry for producing a variety of permanent implants and medical tools, such as hip, knee and shoulder joint prostheses; bone plates; pacemaker casings; screws; nails (Fig. 1).

In orthopaedics, one of the most widely used titanium biomaterials is alloy Ti6Al4V ELI (Extra Low Interstitial) which is characterized by slightly higher hot-workability and brittle crack resistance than the commercial alloy Ti6Al4V owing

Fig. 1. Examples of medical applications of Grade 5 Titanium ELI [3, 12, 23]

to lower oxygen and iron content in its chemical composition. Ti6Al4V ELI alloys have high deformation resistance and low thermal conductivity, which makes hot and cold working difficult [1, 19]. Numerous works on forming titanium and its alloys were published in the last 10 years. However, due to discrepancies in the reported results, it is difficult to establish standard forging conditions for this metal and determine the relationship between the forging temperature and microstructure quality. Most of the studies concern the commercial titanium alloy Ti6Al4V, primarily intended for the aircraft industry [6], while there are significantly fewer publications on the forming of alloy Ti6Al4V ELI (Grade 5 ELI) used for medical applications [1, 4, 10–11, 14, 17–19, 22]. In light of the above, it seems justified to investigate the effect of forging temperature on the microstructure and hardness of titanium alloy grade 5 ELI. The results will be used to determine hot-working temperature that ensures obtaining optimum microstructure. It should be mentioned that the research addresses the problem signalled by medical products manufacturers concerning the forging of titanium alloy Ti6Al4V (Grade 5 ELI) on standard hydraulic presses, using electric furnaces for preheating.

METHODS

The tests were performed on titanium alloy grade 5 (Ti6Al4V) ELI. This biomaterial is a two-phase (α+β) martensitic alloy, and its chemical composition is listed in Table 1. The microstructure of this alloy is a composition of α phase and 5 to 25% β phase. The phase change α+β-> β starts at approx. 887°C and end at 985°C [13].

The hot-workability of these alloys after heat refining is higher than that of single-phase α alloys [1, 19]. It should be mentioned that properties of two-phase α+β alloys depend on the kind and percentage of impurities, alloying elements as well as the content of individual phases. Their strength increases with increasing the β phase content. The mechanical properties of titanium alloy Ti6Al4V ELI are listed in Table 2. Shown in Fig. 2, test specimens had a diameter of 8 mm and a length of 10 mm and were subjected to commercial annealing for 60 minutes at a temperature of 800°C [20].

The study involved performing open die forging of titanium alloy grade 5 ELI by the method described in the standard PN-H-04411:1983. This method is widely used for metal products and semi-finished products, primarily to

Table 1. Chemical composition of Grade 5 Titanium ELI

Al	V	O	C	H	Fe	Ti
6.42	4.19	0.101	0.011	0.0052	0.198	Balance

Table 2. Mechanical properties of titanium alloy grade 5 ELI

Tensile strength R_m [MPa]	Proof stress $R_{0.2}$ [MPa]	Elastic modulus x 10^3 [MPa]	Elongation A_5 [%]	Reduction in area Z [%]
>900–1160	>830	95	8 -10	>25

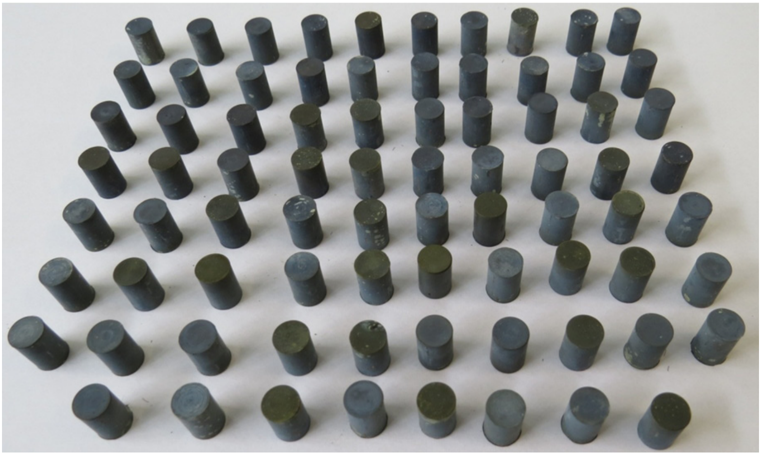


Fig. 2. Specimens made of titanium grade 5 ELI

determine their workability in given thermal conditions and to detect surface and subsurface defects during deformation. The range of forging temperature for Titanium Grade 5 ELI was selected based on an overview of the literature [1, 3–5, 11–19, 22–23] and set to 750°C÷1100°C. As recommended by the standard, the tests consisted in open-die hot forging of a batch of specimens (3 specimens per each temperature changed every 50°C) to 1/3 of their initial height in a specially designed open-die upsetting device (Fig. 3) mounted in the hydraulic punching machine Nargesa MX-700.

The temperature of the dies was maintained at 200 °C and controlled with the Ebro TFI 650 infrared thermometer. The press was provided with a measuring system enabling determination of variations in the force parameters during the forging process. The measuring system comprised a measuring card, pressure and displacement sensors and was provided with the „Samba" software. Pressure in the hydraulic unit was measured with the „Samba" controlled measuring card. The following were measured during the experiments: workpiece compression time, pressure and tool travel. The results were used to determine relationships between yield stresses and equivalent strains [8, 24].

The experimental flow curves and curves obtained from the literature served for designing a model of titanium alloy Ti6Al4V ELI for numerical analysis. The numerical modelling of upsetting titanium grade 5 ELI was performed using the process simulation software Deform 3D [7]. The forging temperature was set to 750°C to simulate real process forging conditions.

To determine the effect of forging temperature on the microstructure of titanium alloy Grade 5 ELI, the reference specimens and those produced by open-die forging under the applied temperature ranges were subjected to microstructural examination. To this end, metallographic specimens were prepared in accordance with the following procedure [2, 25]:

The specimens were cut along the symmetry line using the Struers Secotom with the application of water cooling and pre-treatment, i.e., test specimen grinding and mounting in Epidian 5 resin.

The mounted specimens were ground and polished using a Buehler grinder and polisher and abrasive discs with three different gradations set to 80, 200 and 400, respectively. Following the

Fig. 3. Hydraulic press with upsetting device

grinding process, the specimens were subjected to polishing for 10 minutes with SiO2 suspension using a 0.05 μm disc.

The specimens were subjected to etching by approx. 2 seconds with Kroll's reagent.

Figure 4 shows metallographic specimens for microstructural examination.

The examination of initial and post-forging microstructure of alloy Grade 5 specimens in the zones indicated in Fig. 4 was made by optical microscopy. The microstructural examination was performed using the Nikon MA200 for capturing images in brightfield mode.

Similarly to microstructural examination, the hardness of metallographic specimens was measured in the zones indicated in Fig. 4. The measurements were made using a Future-tech FM800 tester by the Vickers method in compliance with the methods described in PN-EN ISO 6507–1:2006, under a load of 98.07 N [21]. The main aim of the measurements was to determine the effect of forging temperature on the hardness of Titanium Grade 5 ELI.

RESEARCH RESULTS AND ANALYSIS

Following the upsetting process under varying thermal conditions, the titanium Grade 5 ELI specimens had their surface visually inspected. Specimens showing the presence of defects such as cracks, delamination and other signs of material coherence loss were rejected. Surface defects examined for their type or depth were then ground using an abrasive disk. Table 3 compares the results of visual valuation of select specimens deformed in the temperature range 750°C÷1100°C in compliance with the standard PN-H-04411:1983, and provides a description of defects on the end face and flank of upset specimens made of titanium grade 5 ELI.

The results were used to determine the relationship between yield stresses and strains of titanium alloy grade 5 ELI for the applied temperatures; the relationship is shown in Fig. 5. Analysing the experimental flow curves it can be observed that the yield stresses decrease with an increase in the forging temperature.

Fig. 4. Metallographic specimens for microscopic examination; red colour marks zones examined for microstructure quality and hardness

Table 3. Surface of titanium grade 5 specimens after upsetting

<table>
<tr><td colspan="6">750°C</td><td colspan="6">800°C</td><td colspan="6">850°C</td><td colspan="6">900°C</td><td colspan="6">950°C</td></tr>
<tr><td colspan="6"></td><td colspan="6"></td><td colspan="6"></td><td colspan="6"></td><td colspan="6"></td></tr>
<tr><td colspan="30">Correct specimens – no delamination, cracks or dye penetration.</td></tr>
<tr><td colspan="10">1000°C</td><td colspan="10">1050°C</td><td colspan="10">1100°C</td></tr>
<tr><td colspan="10"></td><td colspan="10"></td><td colspan="10"></td></tr>
<tr><td colspan="30">Rejected specimens – no delamination and cracks; visible white oxide spots on specimen end face and flank (caused by heavy oxidation of titanium alloy at elevated temperatures, this leads to a significant reduction in its fatigue strength, which is undesired in the case of implants), local burn-through caused by too high forging temperature.</td></tr>
</table>

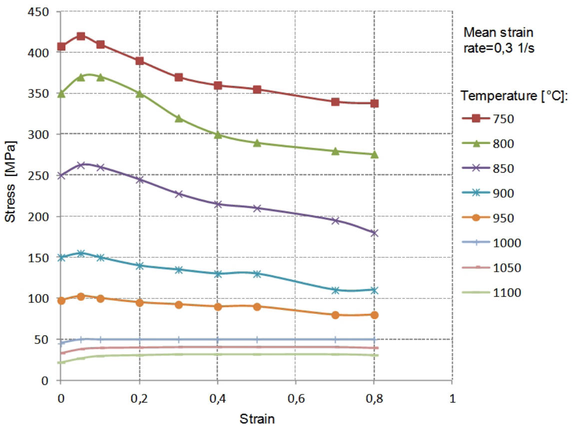


Fig. 5. Yield stresses versus strains for titanium alloy grade 5 ELI in annealed state

Figure 6 illustrates the effective strains and temperatures in the cross section of an upset specimen deformed at a temperature of 750°C, obtained in the numerical simulations by Deform 3D. The highest effective strains occur in the centre of the upset specimen, while the lowest values of effective strain can be observed at the below workpiece-tools contact surface. Analysing the distribution of temperature in the deformed specimen it can be observed that the highest temperatures occur on the workpiece flanks, where the metal flows freely and has no contact with the tools. The lowest temperatures can be observed on the end face of the workpiece, where the metal undergoes cooling due to contact with the much colder tools. The difference in temperatures in individual regions of the product can range 100÷160°C.

The microstructure of reference samples is shown in Fig. 7. The microstructure of the base material is a composition of a reinforced α phase solution and a ≤10% β phase solution. After full annealing, the material exhibits a globular equiaxial microstructure with middle-size grain. The microstructure examination reveals the presence of α phase grains and β phase grains predominantly on the boundary of the α phase grains.

The microstructure of the biomaterial subjected to forging at a temperature of 750°C is uniform (Fig. 8). Figure 8a illustrates the direction of metal flow during the forging process. It must be noted that the α phase that prevails in the microstructure of titanium alloy grade 5 deformed at the temperature of 750°C, as can be seen in Figure 8c. On the other hand, Figure 8b reveals a fine-grained structure (below 10 µm). After forging and full annealing, most grains in the central part of the specimen have equiaxial shape.

The microstructure of the specimens subjected to forging in the temperature range between 800°C and 900°C (Fig. 9÷11) can be described as similar to the microstructure obtained after the forging process at a temperature of 750°C. They are characterized by a relatively uniform, fine-grained structure, primarily comprising the α phase with equiaxial globular grains. The use

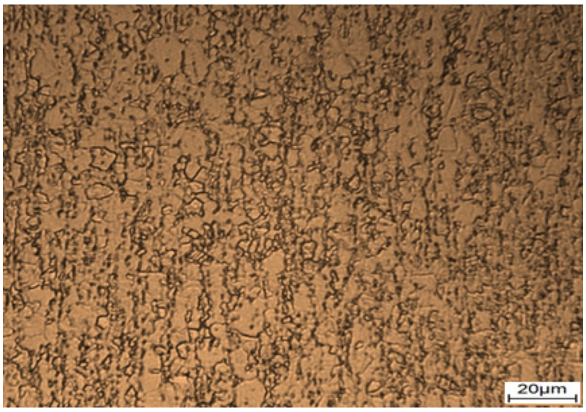


Fig. 7. Microstructure of titanium Ti-6V-4Al ELI specimens prior to forging

a) b)

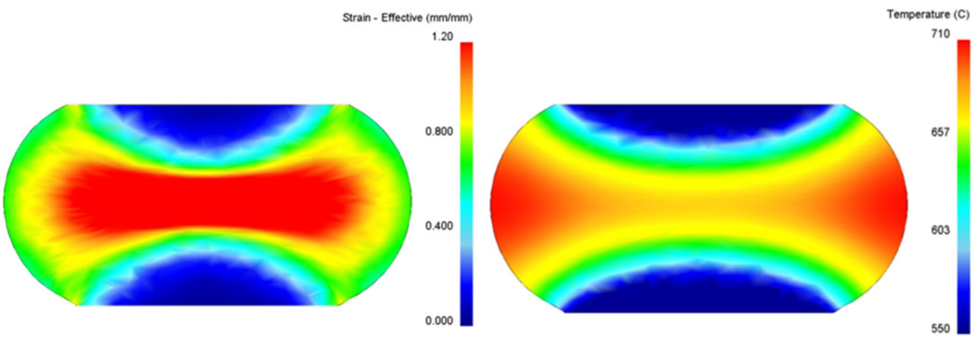


Fig. 6. Effective strains (a) and temperatures (b) in upset product deformed at 750°C numerically simulated by DEFORM 3D

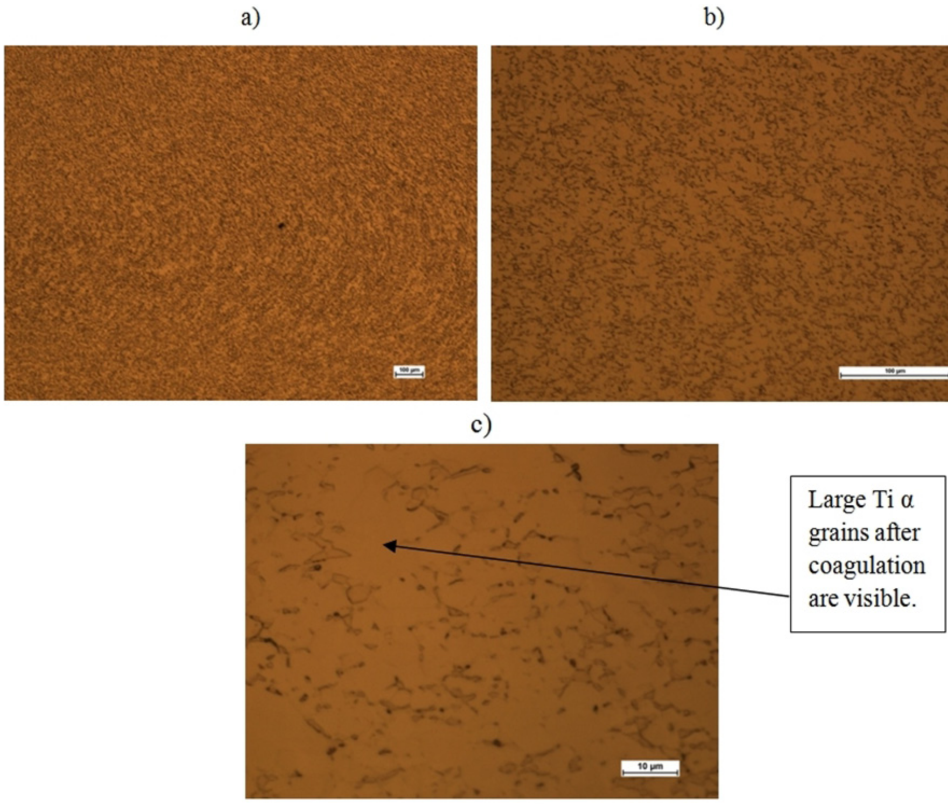


Fig. 8. Microstructure of titanium Ti-6V-4Al ELI after deformation at 750°C and annealing (centre of specimen; 3 magnified images)

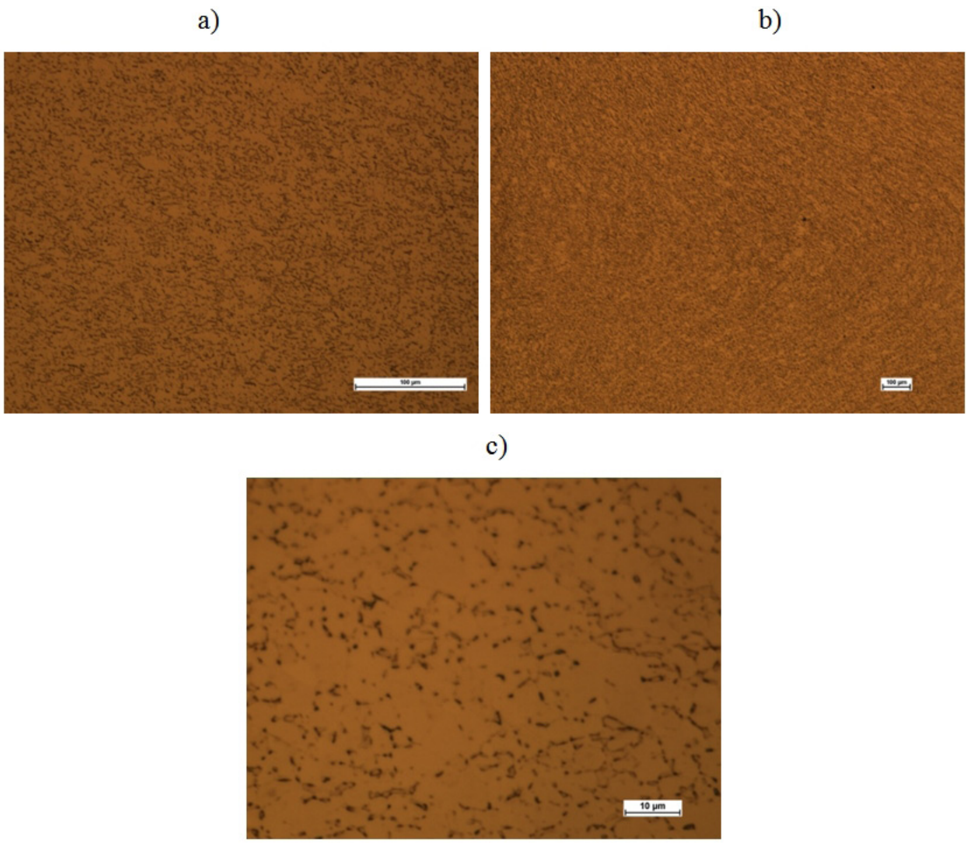


Fig. 9. Microstructure of titanium Ti-6V-4Al ELI after deformation at 800°C and annealing (centre of specimen; 3 magnified images)

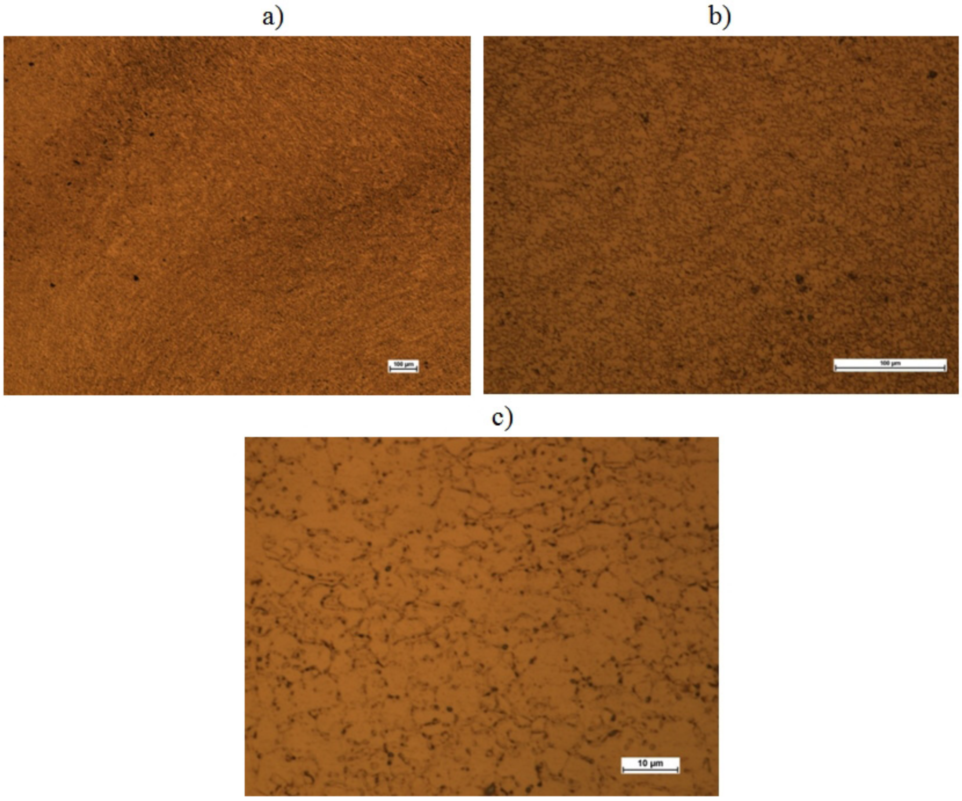


Fig. 10. Microstructure of titanium Ti-6V-4Al ELI after deformation at 850°C and annealing (centre of specimen; 3 magnified images)

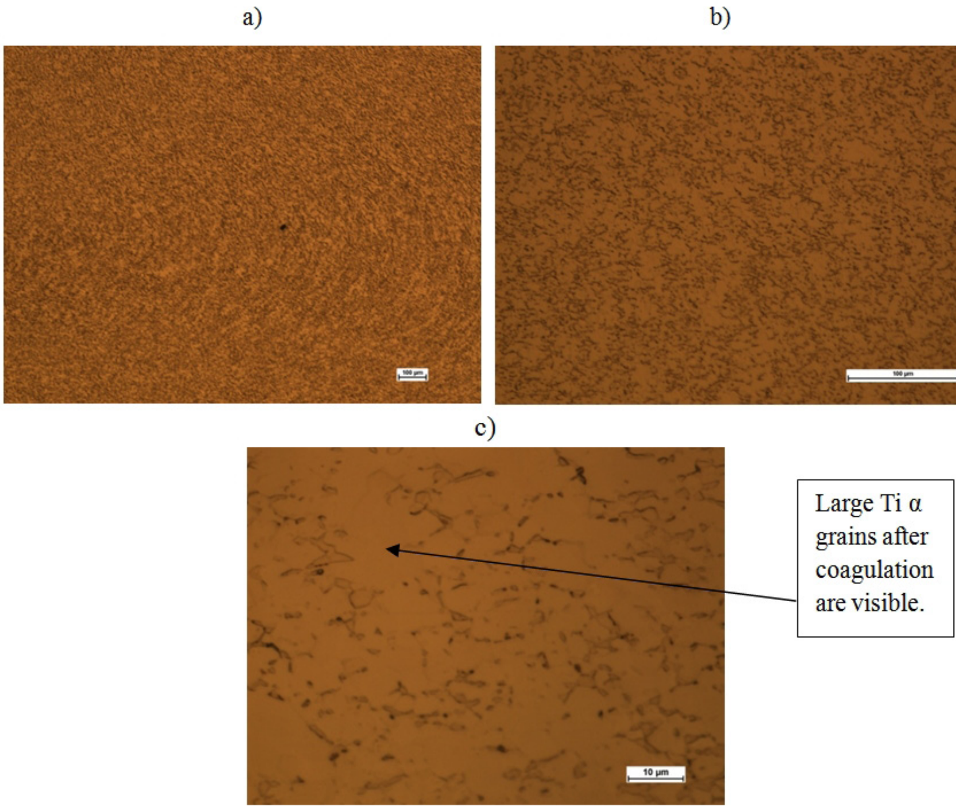


Fig. 11. Microstructure of titanium Ti-6V-4Al ELI after deformation at 900°C and annealing (centre of specimen; 3 magnified images)

of metal forming in conditions of the α+ β -> β transformation temperature requires the application of higher forming forces, but the produced structure is homogenous to a large extent.

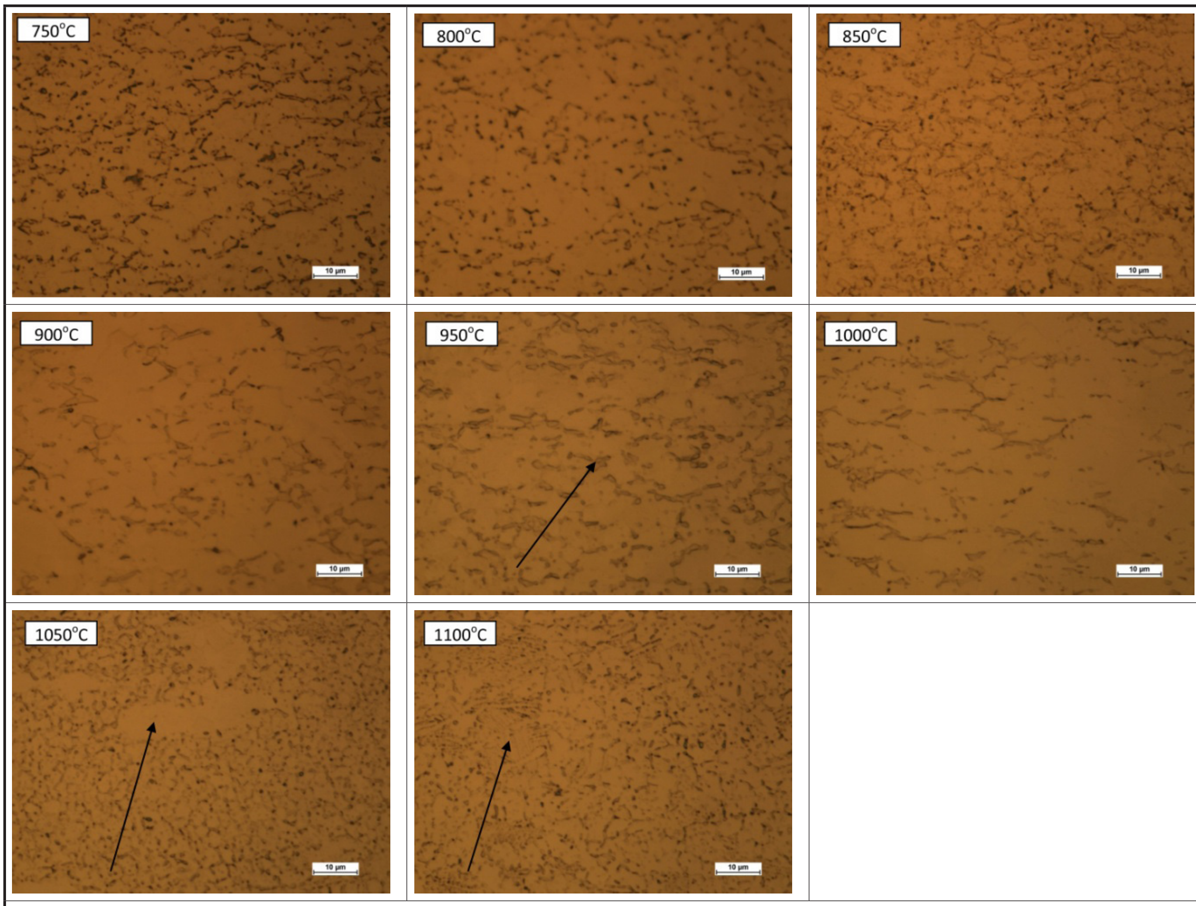
In the temperature range between 900°C and 1000°C the α grains gradually increase. At the temperature of 1050°C one can observe a clear coagulation of the α phase grains. The produced microstructures are compared in Table 4.

At the temperature of 1100°C one can notice big grains with a lamellar structure α and β that fill in the original β grains (Tab. 4). The nucleation and growth of α grains are perpendicular to the boundary of the original β grain and perpendicular to the growing colony that forms Widmanstätten patterns. On the boundaries of former β grain one can notice chain-like α grains.

Figure 12 shows the microstructures of titanium Ti-6V-4Al ELI deformed at different temperatures below the tool-workpiece contact surface. The microstructure of specimens deformed in the temperature range 750÷1000°C is homogenous and oriented at a direction of metal flow. The figure shows an example of microstructure deformed at a temperature of 750°C. After the forging process at 1050°C and 1100 °C and annealing, the region at the outer surfaces of the upset specimen is characterized by a coarse-grained structure.

The lamellar structures at the surface consisting of alternately arranged α and β-phase lammellas fill the transformed grain of the β phase and significantly differ from the equiaxial structures observed in the specimen centre. On the grain boundary one can observe quite big chain-like release of the α phase. The nucleation and growth of α grain is perpendicular both to the boundary of primary β grain and to the growing colony of Widmanstätten patterns.

Table 4. Comparison of microstructure of titanium grade 5 after hot open-die forging (centre of upset specimen)



Arrows indicate:

- 950°C: Visible chain-like coagulated α-secondary Ti grain on the boundaries of globular Ti α grain.
- 1050°C: Above: Large Ti α grains after coagulation are visible. The micro-structure greatly varies in terms of grain size.
- 1100°C: Bimodal microstructure with visible colonies of α` and β strips (Widmanstätten patterns). They are caused by forging at a temperature above the ->β transformation point and slow cooling at a rate lower than the critical one. It is characterized by the presence of parallel lamellas that fill the areas of the original β grain. Lamellas are rather thick because the cooling process was slow. Local transformation only, so the rest consists of the α phase grain.

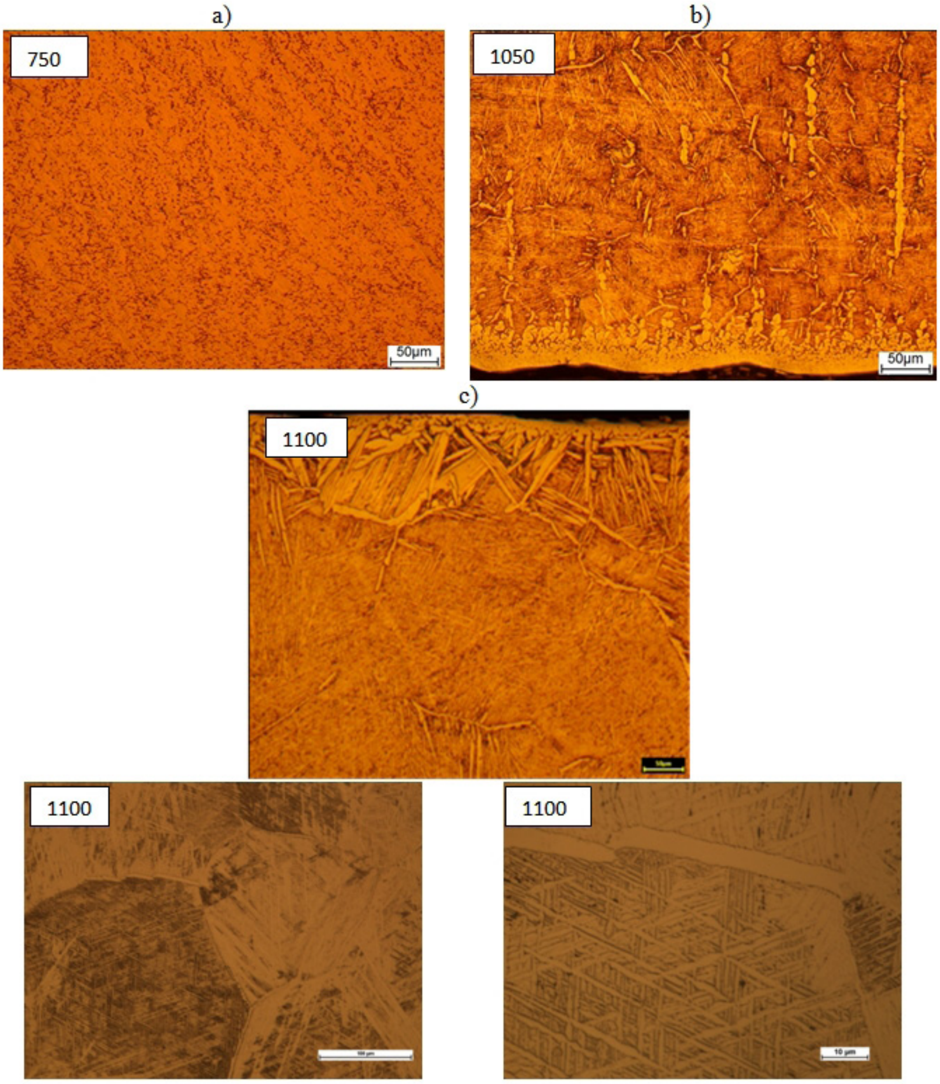


Fig. 12. Microstructure of titanium Ti-6V-4Al ELI after deformation at 750°C (a); 1050°C (b); 1100°C (c) (below tool-workpiece contact surface

It should be mentioned that the globular structures occurring in the temperature range from 750°C to 1050°C have higher strength and ductility. On the other hand, the lamellar structures visible at the temperature of 1100°C have a higher crack resistance. Increasing the temperature leads to a gradual growth of the grain. After the forging process at a temperature higher than 950°C, we can observe considerable heterogeneity of the specimen structure. The central region of the specimens characterized by the highest degree of deformation is predominantly an equiaxial, fine-grained structure, however the accompanying coagulated α phase grains and the α +β lamellar structure-filled transformed β grains reveal a considerable structural heterogeneity in terms of grain size and shape. As a result of the application of forging after annealing in the range α+β and then annealing, the most homogenous and fine-grained structure is produced if the forging temperature does not exceed 850–900°C.

Figure 13 illustrates the relationship between hardness and temperature for two measuring zones of deformed titanium grade 5 ELI.

Analysing the hardness results by the Vickers method it can be observed that mean hardness below the tool-workpiece contact surface is higher than that occurring in the specimen centre where the effective strains are the highest. This results from the fact that the metal undergoes hardening below the tool-workpiece contact surface, which is caused by rapid cooling of titanium alloy due to contact with the colder tools and the presence of high friction forces.

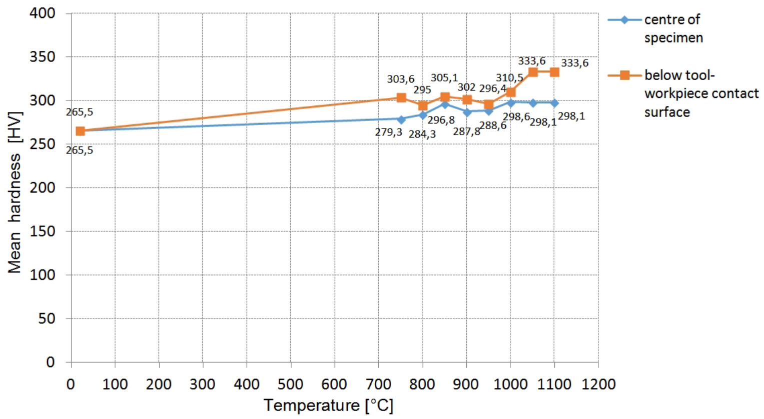


Fig. 13. Mean hardness of titanium grade 5 ELI during deformation at 750÷1100°C and prior to deformation, at 20°C

CONCLUSIONS

The results have led to formulation of the following conclusions:

- Following recrystallization annealing at the temperature of 800°C, the forging process in the temperature range between 750°C and 900°C and then stress relieving at 600°C, the microstructure of titanium alloy grade 5 ELI is fine-grained and homogenous to a great extent. In the central, highest-deformed region of the specimen, a fine-grained equiaxial structure prevails. Despite lower yield stresses, the microstructure of the titanium alloy specimens deformed at the temperature exceeding 950°C is considerably heterogeneous. The lamellar structures at the surface contained in the transformed β-phase grains significantly differ from those observed in the central region of the equiaxial specimens.
- The experimental results and literature survey have revealed that the microstructure and hardness of titanium alloy grade 5 ELI significantly depend on the forging temperature, strain value and rate as well as the rate of cooling after deformation. The changes in the microstructure and phase composition observed in different regions of the upset product result from high sensitivity of two-phase titanium alloys to deformation temperature, strain size and rate. By applying suitable values of these factors, i.e., by control of changes in the thermal and mechanical state of the deformation zone, it is possible to change and shape to a great extent mechanical and functional properties of products made of this material.
- Due to low thermal conductivity of titanium alloys and the high friction factor between the tested titanium alloy and the dies, the produced strains are not uniform; in addition, the structure and mechanical properties are not uniform either. The non-uniform distribution of the friction factor over the contact surface leads to the occurrence of zones of varied deformability in the workpiece. Specifically, a limited deformability zone occurs under the surface of the dies, leading to production of a coarse grain structure with lower deformability and strength properties. The strains in other deformation zones are not uniform either, so the produced structure is heterogeneous.
- The forging process brings about heat change and thus an increase in the temperature of the metal. The thermal effect is most visible in the zones of intensive deformation (centre of the workpiece). The difference in the temperatures of different regions of the workpiece can amount to 100÷150°C, which undoubtedly affects the microstructure of the tested titanium alloy.
- By establishing the relationship between yield stresses and strains of titanium alloy grade 5 ELI, we could determine the effect of the forging temperature on the stress value. With increasing the forging temperature, the yield stresses decrease.
- The mean hardness of the deformed titanium alloy grade 5 ELI in the central region of the workpiece ranges 279.3÷298.6 HV, while on the surface it is 295÷333.6 HV. The increase in hardness of the deformed titanium alloy on the workpiece-tools contact surface is due to the fact that the metal undergoes rapid cool-

ing as a result of contact with the much colder tools and the occurrence of high friction forces. Consequently, this region of the workpiece becomes harder.

REFERENCES

1. Adamus J. Analysis of forming of titan products by cold forming methods., (in Polish: Analiza kształtowania wyrobów tytanowych metodami obróbki plastycznej na zimno. Series of monographs, 174, Czestochowa University of Technology, Częstochowa, 2010.
2. Bienias J., Mania R., Jakubczak P. and Majerski K. The issues of manufacturing geometrically complicated elements using FML laminates. Composites theory and practice, 4(15), 2015, 243–249.
3. Brunette D.M., Tengvall P., Textor M. and Thomsen P. Titanium in medicine. Springer, Germany, 2001.
4. Ding R., Guo Z.X. and Wilson A. Microstructural evolution of a Ti–6Al–4V alloy during thermomechanical processing. Materials Science and Engineering A, 327, 2002, 233–245.
5. Donachie M.J. Titanium: A Technical Guide, 2nd Edition. ASM International, USA, 2000.
6. Dziubińska A., Gontarz A., Dziubiński M. and Barszcz M. The forming of magnesium alloy forgings for aircraft and automotive applications. Advances in Science and Technology Research Journal, 31 (10), 2016, 158–168.
7. Dziubinska A. and Gontarz A. Limiting phenomena in a new forming process for two-rib plates. Metalurgija, 3(54), 2015, 555–558.
8. Dziubinska A. and Gontarz A. A new method for producing magnesium alloy twin-rib aircraft brackets. Aircraft Engineering and Aerospace Technology, 2(87), 2015, 180–188.
9. Fujibayashi S., Neo M., Kim H.M., Kokubo T. and Nakamura T. Osteoinduction of porous bioactive titanium metal. Biomaterials, 25, 2004, 443–450.
10. Gontarz A., Winiarski G. Numerical and experimental study of producing flanges on hollow parts by extrusion with a movable sleeve. Archives of Metallurgy and Materials, 3(60), 2015, 1917–1921.
11. Gupta R.K., Kumar A. V., Kumar P. R. Effect of Variants of Thermomechanical Working and Annealing Treatment on Titanium Alloy Ti6Al4V Closed Die Forgings. Journal of Materials Engineering and Performance, 25 (6), 2016, DOI: 10.1007/s11665–016–2110–8.
12. Hermawan H., Ramdan D. and Djuansjah J. R. P. Metals for Biomedical Applications. Biomedical Engineering – From Theory to Applications. Edited by Prof. Reza Fazel, Publisher InTech, 2011, 411–430.
13. Hirano T., Murakami T., Taira M., Narushima T. and Ouchi C. Alloy design and properties of new a + b titanium alloy with excellent cold workability, superplasticity and cytocompatibility. ISIJ International, 47, 2007, 745–752.
14. Lampman S. Wrought Titanium and Titanium Alloys. ASM Handbook, Properties and Selection: Nonferrous Alloys and Special – Purpose Materials, ASM International, USA, 1992.
15. Niinomi M., Nakai M., and Hieda J. Development of new metallic alloys for biomedical applications. Acta Biomaterialia, 8, 2012, 3888–3903.
16. Narushima T. Titanium and its alloys as biomaterials. Journal of Japan Institute of Light Metals, 2005, 55, 561–565.
17. Rojas-Olmos D. C., López-Perrusquia N., Doñu-Ruiz M. A., Torres San Miguel C. R. Study Microstructure and Mechanical Properties of Prosthesis of Forging. MRS Online Proceeding Library Archive 1766, 2015, DOI: 10.1557/opl.2015.406.
18. Saitova L.R., Höppel H.W., Göken M., Semenova I.P., Raab G.I. and Valiev R.Z. Fatigue behavior of ultrafine-grained Ti–6Al–4V "ELI" alloy for medical applications. Materials Science and Engineering: A, 503, 2009, 145–147.
19. Sińczak J. Advanced forging technologies high-melting materials (in Polish: Zaawansowane technologie kucia materiałów wysokotopliwych). ARBOR FP Kraków, Kraków, 2013.
20. Swic A., Draczew A. and Gola A. Method of achieving accuracy of thermo-mechanical treatment of low-rigidity shafts. Advances in Science and Technology Research Journal, 29(10), 2016, 62–70.
21. Szala M. and Kot E. Influence of repainting on the mechanical properties, surface topography and microstructure of polyester powder coatings. Advances in Science and Technology Research Journal, 2(11), 2017, 159–165.
22. Tofil A., Tomczak J., Bulzak T. Comparative Analysis of Forging Rolling and Cross-Wedge Rolling of Forgings from Titanium Alloy Ti6Al4V. Key Engineering Materials, 687, 2016, 141–148.
23. Wang K. The use of titanium for medical applications in the USA. Materials Science and Engineering A, 213, 1996, 134–137.
24. Wojcik Ł., Lis K. and Pater Z. Plastometric tests for plasticine as physical modelling material. Open Engineering, 1(6), 2016, 653–659.
25. Wolszczak P. and Cechowicz R. Examination of the influence of shear micro geometrical properties on transverse elasticity the modulus of roving composite materials used in critical constructions. 32nd Riso International Symposium on Materials Science – Composite materials for structural performance: towards higher limits, Denmark, 2011, 487–496.