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**THE KINETICS OF PHASE TRANSFORMATIONS DURING CONTINUOUS COOLING OF Ti6Al4V ALLOY FROM THE DIPHASE  $\alpha + \beta$  RANGE****KINETYKA PRZEMIAN FAZOWYCH PRZY CHŁODZENIU CIĄGŁYM STOPU Ti6Al4V Z ZAKRESU DWUFAZOWEGO  $\alpha + \beta$** 

The paper reports on a study of the kinetics of phase transformations during continuous cooling of the diphasic Ti6Al4V titanium alloy from the diphasic  $\alpha + \beta$  range (950°C). It has been found that within the whole range of applied cooling rates a diffusional  $\beta \rightarrow \alpha$  transformation occurred. Metallographic observations of the alloy's microstructure gave evidence of changes in the  $\alpha$  phase morphology. Upon cooling at between 7.1 and 0.9°C/s the  $\alpha$  phase precipitates show lamellar character (Widmannstätten structure), while at the slowest rates (0.23÷0.011°C/s) the  $\alpha$  phase precipitates in a form of grains. Besides microstructural changes, dilatometric effects were also observed which corroborates occurrence of phase transformations in the alloy.

*Keywords:* phase transformations, microstructure, hardness, dilatometric curve, CCT diagram

W artykule dokonano oceny kinetyki przemian fazowych przy chłodzeniu ciągłym dwufazowego stopu tytanu Ti6Al4V za pomocą wykresu CTPc, wykonanego dla wybranej temperatury 950°C (z zakresu dwufazowego  $\alpha + \beta$ ). Wykazano, że w całym badanym zakresie szybkości chłodzenia stopu od tej temperatury obserwuje się występowanie przemiany dyfuzyjnej  $\beta \rightarrow \alpha$ . Obserwacje metalograficzne stopu Ti6Al4V, chłodzonego z zakresu dwufazowego z różnymi szybkościami, pozwalają zauważyć wyraźne zmiany w morfologii fazy  $\alpha$ . Przy chłodzeniu z szybkościami w zakresie 7,1÷0,9°C/s, wydzielenia fazy  $\alpha$  mają charakter płytkowy (w układzie Widmannstättena), zaś przy najmniejszych szybkościach (0,23÷0,011°C/s) obserwuje się wydzielenia tej fazy w formie ziarnistej. Potwierdzeniem przemian fazowych zachodzących podczas chłodzenia stopu są nie tylko zmiany zachodzące w mikrostrukturze, ale również efekty dylatacyjne obserwowane na dylatogramach chłodzenia.

**1. Introduction**

The recent trend in modern materials engineering is to search after new materials having properties desired by end users, but a lot of effort is also dedicated to improve materials which already exist. The widely applied heat treated ferrous matrix alloys still belong to those having very good mechanical strength and ductility. Alloys with a titanium matrix are currently ones of the most attractive materials, since they are characterised by an excellent combination of specific strength, high temperature creep resistance as well as resistance to corrosion. Titanium alloys are most widely used in the transportation (mainly aircrafts), shipbuilding, chemical, food, electric/electronic, pulp and paper, medical and sport equipment industries, as well as in geology [1÷3]. Due to good biocompatibility (as compared to other metallic biomaterials), titanium alloys are also applied as prosthetic materials, to produce knee and hip

replacement joints, in dentistry and traumatology [4÷7]. They are also applied for parts of steam turbines, jet engines, cars, ships, aircraft covers, and for building of modern submarines, where not only their high relative strength and corrosion resistance are utilised but also para magnetism, which renders difficult such submarine detection by magnetic methods [1]. Unique properties of titanium alloys provide opportunities for improving technological processes and products in various industrial and economic branches.

A typical example of diphasic martensitic titanium alloys is the Ti6Al4V alloy, which is suitable to both plastic working and heat treatment whereby its macrostructure and mechanical properties can widely be varied [1, 2, 8]. Thorough qualitative and quantitative studies of phase transformations and their influence on mechanical properties of the Ti6Al4V alloy are still lacking in literature, although better understanding of phase transformation kinetics during cooling from either  $\alpha + \beta$

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or  $\beta$  range seems essential. Therefore the main objective of the present work was to determine the kinetics of phase transformations occurring during continuous cooling of the Ti6Al4V alloy from 950°C ( $\alpha + \beta$  range).

## 2. Experimental procedure

The Ti6Al4V alloy used in the present work was delivered in a form of a  $\varnothing 35 \times 250$  mm rolled and annealed bar by BIBUS METALS AG. Its chemical composition is given in Table 1.

TABLE 1  
Chemical composition of the Ti6Al4V alloy

Chemical composition, % weight							
Ti	Al	V	Fe	C	O <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub>
bal.	6.5	4.4	0.15	0.023	0.130	0.017	0.0024

The annealing cycle consisted in heating to 750°C, holding at 750°C for 2 hours, furnace cooling to 600°C, holding at 600°C for 2 hours and cooling in air to room temperature.

Small  $\varnothing 4 \times 25$  mm specimens were cut from the bar parallel to its axis and used to examine phase transformation kinetics during continuous cooling in an optical dilatometer. Prior to the cooling stage the samples were heated at of 3°C/minute to 950°C, held for 20 minutes, and then cooled to room temperature at six different rates decreasing from 7.1 to 0.012°C/s. The solution treatment temperature (950°C) is  $\sim 20^\circ\text{C}$  below the  $\beta$ -transus temperature for the tested alloy. This was confirmed by both the microstructural observations, performed on specimens water-quenched from various temperatures lying within the range 700÷1250°C, and dilatometric studies.

In the LS4 dilatometer changes in the specimen length ( $\Delta L$ ) were recorded on a photographic plate as a function of temperature (T) in order to precisely establish the phase transformation start and finish temperatures.

After cooling the specimens were mounted in duracryl, pre-ground on a magnetic grinder, successively ground on abrasive papers with decreasing grain sizes, and polished on a cloth impregnated with SiC suspension. Prior to microscopic observations on a light AX-IOVERT 200 MAT (ZEISS) microscope, the metallographic sections were etched in either 6 vol.% HF or 2 vol.% HF + 2 vol.% HNO<sub>3</sub> water solutions.

A Vickers hardness test was also performed, using a HPO250 type apparatus with a load of 10kG (98N), and the hardness numbers were included in the CCT diagram.

## 3. Results and discussion

Figure 1 shows a microstructure of a specimen rapidly cooled from 970°C ( $\beta$ -transus temperature), wherein oriented, acicular precipitates of apparently  $\alpha'$  phase, formed as a result of a diffusionless  $\beta \rightarrow \alpha'$  transformation, are clearly seen instead of bright-etching  $\alpha$  phase, which was completely dissolved in  $\beta$  during the 60-minute solution treatment step. The specimen hardness is 385 HV<sub>10</sub>.

Figure 2 shows selected dilatometric curves recorded on cooling from 950°C at rates increasing from 0.011 to 7.1°C/s. The curves well exemplify the effect of cooling rate on the  $\beta \rightarrow \alpha$  transformation range  $T_{\beta \rightarrow \alpha}^s \div T_{\beta \rightarrow \alpha}^f$ , where  $T_{\beta \rightarrow \alpha}^s$  and  $T_{\beta \rightarrow \alpha}^f$  denote the temperature at which  $\alpha$  starts to form on cooling from the diphasic range after a certain incubation period and temperature at which the  $\beta \rightarrow \alpha$  transformation terminates, respectively.

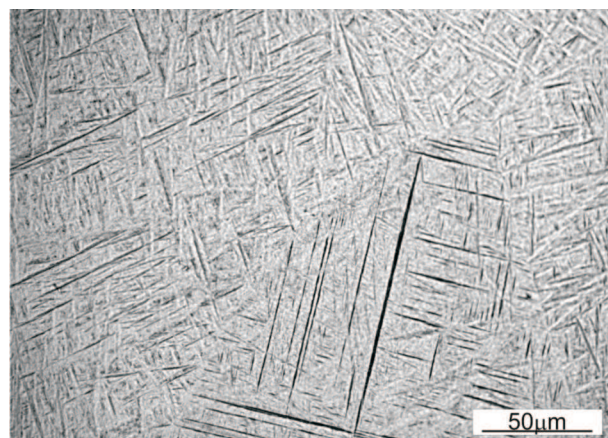
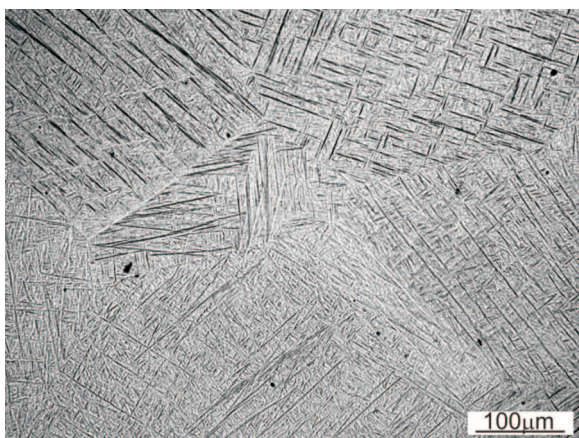


Fig. 1. Microstructures of the Ti6Al4V alloy after heating to 970°C, holding for 60 minutes and cooling in water

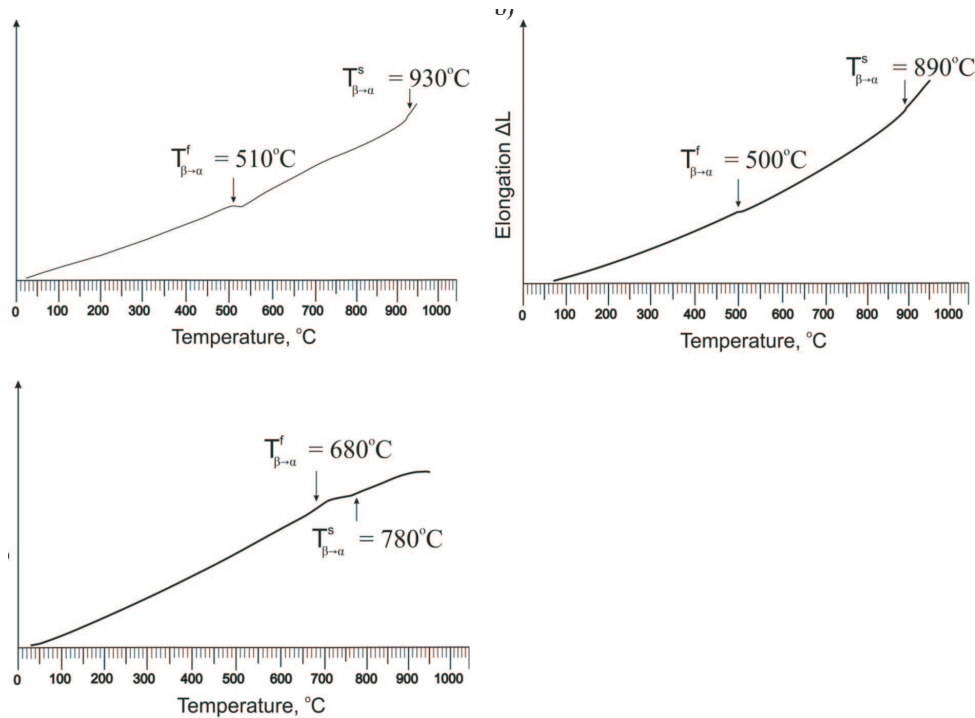


Fig. 2. Dilatometric curves of the Ti6Al4V alloy cooled from  $950^{\circ}\text{C}$  to room temperature at: 0.011  $^{\circ}\text{C/s}$  (Fig. a), 0.23  $^{\circ}\text{C/s}$  (Fig. b) and 7.1  $^{\circ}\text{C/s}$  (Fig. c)

$T_{\beta \rightarrow \alpha}^s$  manifests itself in the dilatometric curves as an accelerated shrinkage of the specimen, while  $T_{\beta \rightarrow \alpha}^f$  is seen as decelerated shrinkage or swelling.

Lowering the solubility of alloy elements in  $\beta$  phase causes the  $\alpha$  phase precipitation. For example the  $\beta \rightarrow \alpha$  transformation start and finish temperatures for cooling at 0.011  $^{\circ}\text{C/s}$  from  $950^{\circ}\text{C}$  are  $T_{\beta \rightarrow \alpha}^s = 930^{\circ}\text{C}$  and  $T_{\beta \rightarrow \alpha}^f = 510^{\circ}\text{C}$ , respectively, as indicated in Fig. 2a.

A thorough analysis of the whole set of six cooling curves permitted drawing a CCT diagram for the Ti6Al4V alloy, which is shown in Figure 3.

The available in literature CCT diagrams for diphas titanium alloys are usually drawn for cooling from the  $\beta$

range [8] and very little, if any, data exists on cooling from the diphas  $\alpha + \beta$  range. The analysis of phase transformation kinetics on cooling from  $950^{\circ}\text{C}$  seems to be justified, because the diphas martensitic titanium alloys are quenched from the  $\alpha + \beta$  field [1, 4, 8], wherein the undissolved  $\alpha$  phase grains prevent grain growth.

As can be seen in Figure 3, a diffusional transformation occurs within the whole range of cooling rates applied, from 7.1  $^{\circ}\text{C/s}$  (air cooling) down to 0.011  $^{\circ}\text{C/s}$ , between the  $T_{\beta \rightarrow \alpha}^s$  and  $T_{\beta \rightarrow \alpha}^f$  lines.  $\alpha_u$  denotes equiaxed  $\alpha$  phase grains which remain undissolved in  $\beta$  at  $950^{\circ}\text{C}$ . The increase in the cooling rate from 0.011 to 7.1  $^{\circ}\text{C/s}$  narrows the transformation range and slightly increases the alloy's hardness, from 283 to 306 HV.

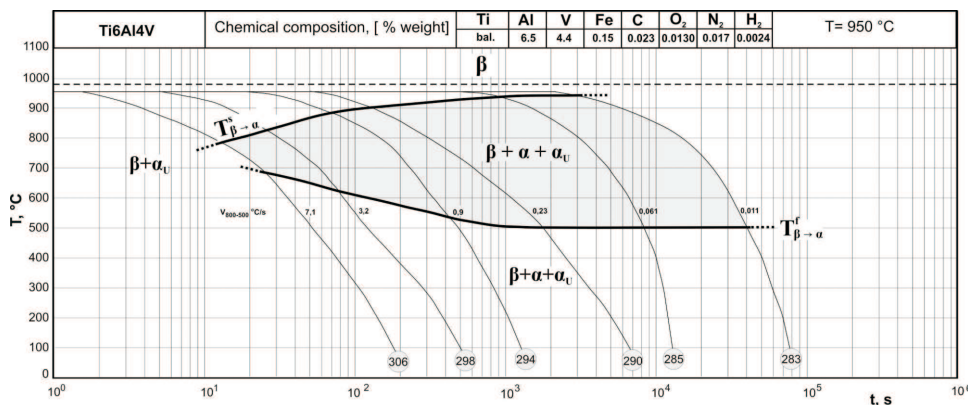


Fig. 3. CCT diagram of the Ti6Al4V alloy

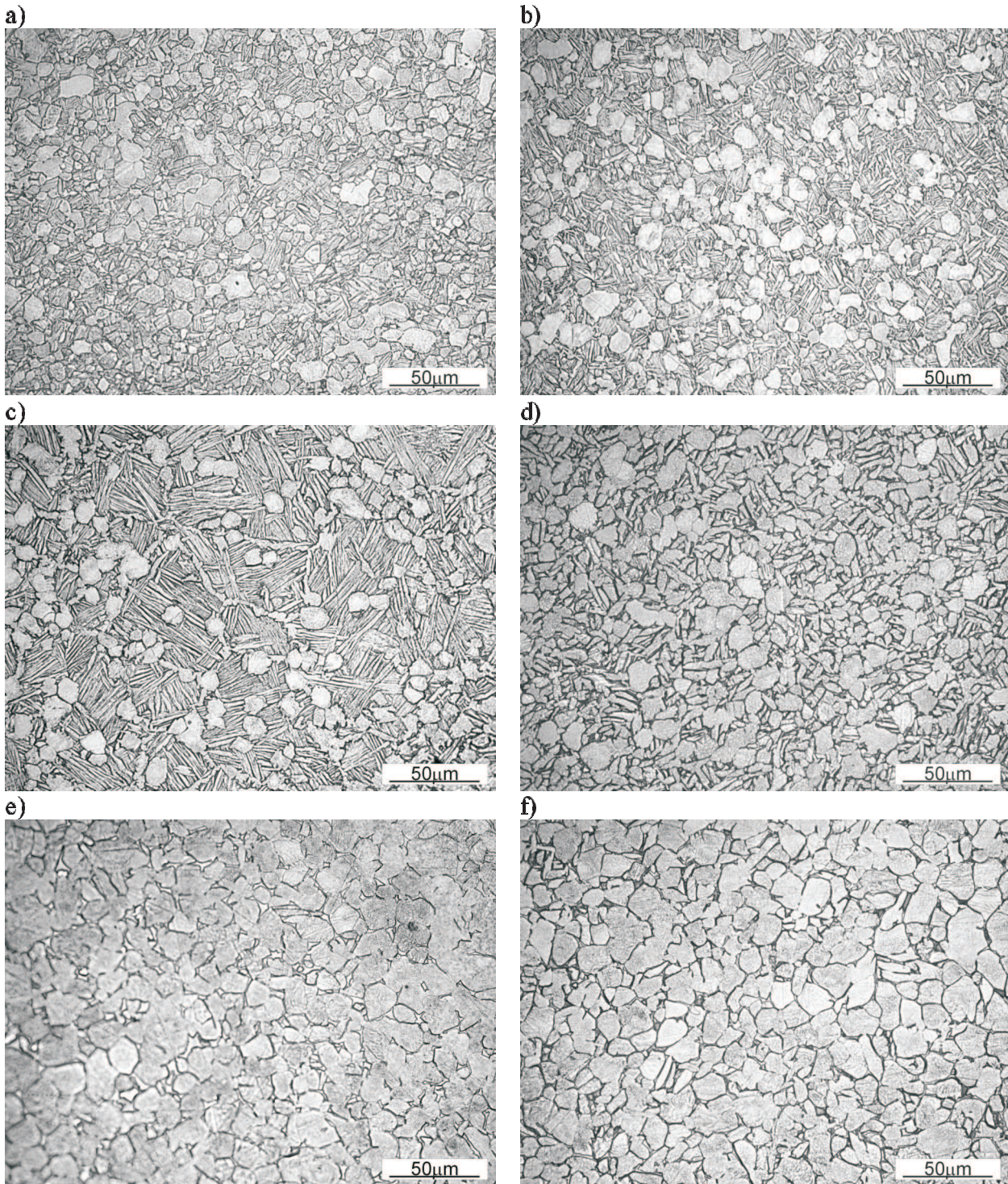


Fig. 4. Micrographs the Ti6Al4V alloy cooled from 950°C at: 7.1°C/s (Fig. a), 3.2°C/s (Fig. b), 0.9°C/s (Fig. c), 0.23°C/s (Fig. d), 0.061°C/s (Fig. e) and 0.011°C/s (Fig. f)

The resulting microstructures are shown in Figure 4. As seen in Figure 4a the  $\alpha$  phase precipitates at three grains contacts and prior  $\beta$  phase grain boundaries. Apart from the undissolved equiaxed  $\alpha$  phase grains, also oriented lamellas of  $\alpha$  phase are formed due to the

diffusional  $\beta \rightarrow \alpha$  transformation. Thus, it should be expected, that the “new”  $\beta$  phase, enriched with  $\beta$  stabilizers, will occur between the  $\alpha$  phase lamellas [1, 2, 4, 8]. After cooling at 7.1°C/s the Ti6Al2Cr2Mo alloy has a hardness of 306 HV and a fine-grained microstructure

with no evidence of titanium martensite  $\alpha'$  although its presence upon cooling in air from 870°C is possible and has been reported in the literature [8].

A decrease in the cooling rate from 7.1 to 3.2 and subsequently to 0.9°C/s (Figs 4b, c) results in a small drop in hardness to 298 and 294 HV, respectively. The lamellas of  $\alpha$  phase precipitate in the Widmannstätten structure [3, 9] forming colonies oriented in the privileged directions within each  $\beta$  grain (Figs 4b, c).

A further decrease in the cooling rate to 0.23°C/s (Fig. 4d) induces a “volumetric growth” of the  $\alpha$  phase changing its morphology from needle-like to equiaxed. The primary  $\alpha$  grains are also clearly seen as at higher cooling rates. Such microstructure gives a hardness of 290 HV.

The application of even lower cooling rates, i.e. 0.061 and 0.011°C/s (Figs 4e, f) apparently increases the grain size suppressing the lamellar form of  $\alpha$ . The darker islands between  $\alpha$  grains (Fig. 4f) are presumably the residue of untransformed  $\beta$ . The hardness numbers of specimens cooled at 0.061 and 0.011°C/s are 285 and 283 HV, respectively.

#### 4. Conclusions and future research

The phase transformation kinetics during continuous cooling from 950°C of the diphasic Ti6Al4V titanium alloy was studied by dilatometry, hardness test and metallographic observations. As a result a CCT diagram was drawn. Additionally the obtained results have demonstrated that:

- the diffusional  $\beta \rightarrow \alpha$  transformation occurs the Ti6Al4V alloy during cooling from 950°C at rates between 7.1 and 0.011°C/s and results in hardness falling into the range 306÷283 HV
- the morphology of  $\alpha$  phase and fineness of the structure depend on the cooling rate. Cooling at 7.1÷0.9°C/s produces a needle-like  $\alpha$  phase (Widmannstätten structure) which become equiaxed, coarse-grained at lower cooling rates.

A more accurate analysis of the phase transformation kinetics taking place in the Ti6Al4V alloy will require additional experiments employing faster cooling rates. The obtained results shall constitute the basis of further work aimed at the analysis and interpretation of phase transformations occurring during tempering (aging) of the Ti6Al4V alloy and construction of the so-called CHT diagram.

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#### REFERENCES

- [1] C. L a y e n s, M. P e t e r s (red.), Titanium and titanium alloys: fundamentals and applications, Wiley-VCH, 2003.
- [2] T. A h m e d, H. R a c k, Phase transformations during cooling in  $\alpha + \beta$  titanium alloys, *Mat. Sci. Eng.* **A243**, 206-211 (1998).
- [3] ASM committee on titanium alloys, Heat treatment of titanium and titanium alloys, *Metals Handbook*, 9<sup>th</sup> edition **4** 763-774 (1981).
- [4] V. A. J o s h i, Titanium Alloys: An atlas of structures and fracture features, 7-13 (2006).
- [5] H. G a r b a c z, P. W i e c i ń s k i, T. W i e r z c h o ń, K. J. K u r z y d ł o w s k i, Effect of surface treatment on the microstructure of Ti6Al4V, *Archives of Metallurgy and Materials* **51** 4, 625-630 (2006).
- [6] S. S e a g l e, L. B a r t l o, Physical Metallurgy and Metallography of Titanium Alloys, *Metal Eng., Quarterly* **8**, 1-10 (1968).
- [7] C. O l i n, Titanium in cardiac and cardiovascular applications. *Titanium in medicine*, Springer, 889-908 (2001).
- [8] H. H. W e i g a n d, Umwandlung von ( $\alpha + \beta$ ) – Titanlegierungen mit Aluminium, “*Metallkunde*” **54** 1, 43-49 (1963).
- [9] E. R o ź n i a t a, The role of cementite in fracture toughness of L200HNM cast steel, Ph. D. Thesis, AGH University of Science and Technology, Krakow, 2008.