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The phase transformations during continuous cooling of Ti6Al7Nb alloy from the two-phase α+β range

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ABSTRACT

Purpose: The phase transformations during continuous cooling from the two-phase α+β range in Ti6Al7Nb alloy has been determined.
Design/methodology/approach: The phase transformations during continuous cooling of investigated alloy was elaborated using an L78 R.I.T.A dilatometer of the German Linseis Company. The microstructure of investigated alloy cooled from the two-phase α+β range was examinated by a light microscope Axiovert 200MAT. The measurements of hardness were performed with the Vickers HPO 250 apparatus.
Findings: The relationship between cooling rate and microstructure morphology, hardness as well as dilatation effects has been determined.
Research limitations/implications: The results will be used for the interpretation of phase transformations occurring at continuous cooling and at tempering from as-quenched state of the investigated alloy. Currently, the investigations of the kinetics phase transformation at continuous heating from as-quenched state will help to performed the Continuous-Heating-Transformation diagrams (CHT). These diagrams show the possibility of the regulation of the progress of successive (intermediate) transformations at tempering and thus influence the final alloy properties, including their hardness and fracture toughness. It is expected that the determination of final mechanical properties by means of regulating the progress of successive transformations occurring during heating from the as-quenched state is possible also in the Ti6Al7Nb alloy.
Practical implications: The obtained results will be applied for the optimization of the heat treatment technology and for achieving the required mechanical properties of the Ti6Al7Nb alloy.
Originality/value: The obtained results, supported in the future by additional cooling curves, will be used for the development of the original, full CCT diagram of the Ti6Al7Nb alloy cooled continuously from the two-phase α+β range.
Keywords: Metallic alloys; Titanium alloy; Phase transformation; Microstructure; Dilatometric analysis

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1. Introduction

Alloys on titanium bases play a significant role in the current material engineering [1-3]. Out of several engineering materials these alloys are singled out-first of all-by a low density, good corrosion resistance, high relative strength at ambient and higher temperatures, relatively low Young's modulus and paramagnetism, which determines their application in industry [1-5].

The beginning of the XXI-st century witnesses an intensive development of titanium alloys belonging to the so-called III-rd generation, which means the β and pseudo β alloys, e.g. Ti12Mo6Zr2Fe, Ti13Nb13Zr, Ti15Mo5Zr3Al, Ti3Al5V6Cr3Mo3Zr [3,4, 6-8]. These alloys are being implemented in medicine, among others for elements of endoprosthesis of hip and knee joints [2, 3, 5, 9]. Currently curried out investigations concern also their eventual application in dental implantation in the future [2,3,7,8]. Earlier, it means at the end of the XX-th century, titanium alloys of the second generation occurred, such as e.g. Ti6Al7Nb. This is a two-phase alloy, due to which there is a possibility of shaping its mechanical properties by plastic working methods and by heat treatments [1,2,9,10]. In the first generation alloy of the Ti6Al4V grade, 4% of V was substituted by 7% of Nb. The substitution of these elements was not only due to differences of their specific gravity but due to the determined applications-e.g. biomedical. Long-term investigations performed in the 70-th of the previous century indicated that Nb is a vital element (tolerated by human organism) in contrast to vanadium causing cytotoxic and carcinogenic reactions in human organisms [3, 5].

Mechanical properties of two-phase alloys are shaped, among others, by heat treatment and plastic working methods [9-14]. Development of the optimal technology of such treatment requires the knowledge of phase transformation kinetics occurring in these alloys under isothermal conditions as well as at a continuous cooling [2, 15-17]. Knowing the kinetics of phase transformations at cooling two-phase titanium alloys is very important in industrial practice, due to the presence, in this temperature range, a certain amount of the α phase preventing grain limits migration and the β phase grain growth [1,2,10,18]. There are several data in the available domestic and foreign literature on the phase transformation kinetics occurring at a continuous cooling of alloys on the ferrous matrix. These are the so-called Bain diagrams characterizing phase transformations occurring in these alloys at a continuous cooling at various velocities [19]. Generally the CCT (continuous-cooling-transformation) diagrams are also known at a continuous cooling for alloys on the titanium bases. These are diagrams developed for the two-phase alloys, e.g. Ti3Al2.5V, Ti6Al4Cr, Ti6Al3Mo1V, Ti6Al6Mo, Ti6Al4V, Ti6Al2Mo2CrFe as well as for single-phase, e.g. Ti6Al2.5Sn [15,16,20].

Investigations of phase transformations occurring at a continuous cooling in the new generation alloy of the Ti6Al7Nb grade are supplementing the above given data. The analysis of phase transformations and changes occurring in the microstructure after cooling this alloy from the annealing temperature from the two-phase range (just below the β-transus temperature). The detailed analysis of phase transformations and microstructures will allow, in the future, the development of the total CHT diagrams, interpretation of the phase transformation occurring at tempering (aging) as well as the development of the optimal heat treatment technology for the Ti6Al7Nb alloy.

2. Research material

Investigations were performed on the two-phase martensitic Ti6Al7Nb alloy. The detailed chemical composition of the alloy according to the ISO 5832-11 as well as the ladle analysis are given in Table 1.

Table 1.
Chemical composition of Ti6Al7Nb alloy according to ISO 5832-11 and heat analysis

	Chemical composition, [wt. %]					
-	Al	Nb	Fe	C	H	O
ISO 5832-11	5.5-6.5	6.5-7.5	max. 0.25	max. 0.08	max. 0.009	max. 0.2
Analysis	5.80	6.50	0.037	0.017	0.001	0.14

The material was delivered in a form of a bar 8 mm in diameter and 1000 mm in length. It was subjected by manufacturer to the hot working in the two-phase α+β range followed by air-cooling.

3. Experimental procedure

Temperatures of phase transformations occurring during cooling of the Ti6Al7Nb alloy were measured by the dilatometric method using an L78 R.I.T.A. dilatometer of the German LINSEIS Company. Sample elongations resulting from the temperature changes were recorded digitally. Samples with dimensions of ∅3x10 mm were heated at of 5°C/minute to 970°C, held for 20 minutes and then cooled to room temperature at four different rates decreasing from 25 to 0.05°C/s. The solution treatment temperature (970°C) is ~40°C below the β-transus temperature for the tested alloy [21]. 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. The cooling curves was differentiated $\Delta L/\Delta T=f(T)$. This makes possible to determine precisely the start and finish temperatures of the phase transformations.

After cooling the samples were mounted in duracryl and mechanically ground on the magnetic grinder with the borazon grinding wheel. Then, they were ground on abrasive papers with gradually decreasing abrasive grains. The ground samples were polished in SiC suspension. Subsequently the samples were etched in two steps:

- 6% HF-for a few seconds,
- solution consisting of 2 ml HF+ 2 ml HNO_3 + 96 ml H_2O-for a few seconds.

Microscopic examinations were performed on the light microscope AXIOVERT 200MAT of the ZEISS Company.
Hardness was measured by the Vickers apparatus, type HPO 250 at the load of 10 kG. Three hardness measurements, from which the arithmetic mean was calculated, were carried out on each sample.

Microhardness of particular phases in as-delivered and in as-quenched conditions was measured on the Hannemann apparatus at the load of 10 G.

4. Results and discussion

The microstructure of the Ti6Al7Nb alloy, obtained after heating to a temperature of 970°C, annealing for 20 minutes and water cooling, is shown in Figure 1. This is a bimodal microstructure containing large, nearly equiaxial, grains of the α phase, which at this temperature were not subjected into the β phase transformation and a certain volume fraction of the β phase called the transformed β phase [1,2,15,16]. The new β phase is fine-grained and was obtained from the β phase, which grains were still not growing at a temperature of 970°C. The macroscopic hardness of the sample, water cooled with a rate of app. 40°C/s from 970°C, equals 315HV.

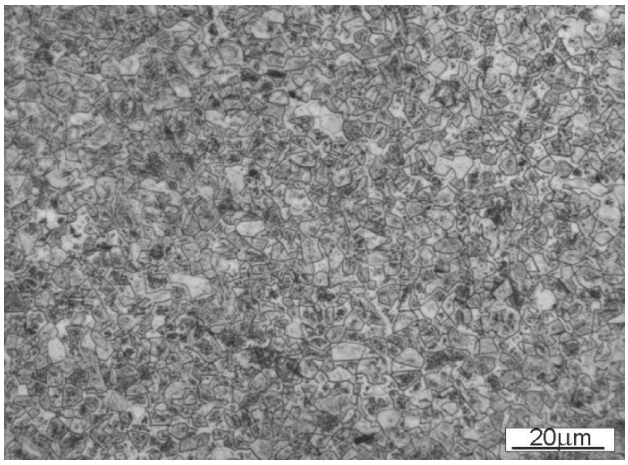

Fig. 1. The microstructure of Ti6Al7Nb alloy after heating to 970ºC, annealing for 20 minutes and water quenching

Schemes of heating dilatometric samples from as delivered condition to a temperature of 970°C, annealing for 1200 s and cooling to a room temperature with a rate of 5°C/s are presented in Figs. 2, 3.

Figure 2 presents the described scheme in the system ΔL=f(T), while Figure 3 in the system ΔL=f(t). These schemes consist of 3 segments: heating (I), annealing (II) and cooling (III). Segment (I) confirms the selection of the proper annealing temperature, even regardless of applying a high heating rate of the dilatometric sample, being 5°C/s. For this heating rate the temperature of the real transformation equals app. 920°C [21], and after its exceeding a strong diffusion of alloying elements to the β phase zone occurs and a shrinkage is observed on the dilatometric curve. Thus, during heating with a rate of 5°C/s to a temperature of 970°C the alloy is still within the two-phase range (Figs. 2, 3). The segment (II) of isothermal annealing deserves attention. During annealing at 970°C the change of the chemical composition of both phases occurs. At a constant annealing temperature the volume fraction of the β phase probably increases and that of the α phase decreases. It should be expected that during the isothermal annealing the phases chemical composition in the moment of the start of this „segment" is different than in the moment of its finish, before cooling (III). It should be noted that such situation occurs also in alloys on the ferrous base, where the austenite chemical composition in the moment of finishing the austenitising and starting cooling is different than at the beginning of this process. The schematic presentation shown in Figure 3 confirms these considerations.

It can be also seen in Figure 3, that the time of heating to a temperature of 970°C equals 180 s, annealing time-1200 s, while the start of the sample cooling occurs after 1380s. After 180 s (in the moment of obtaining the real annealing temperature), the length of the dilatometric sample in relation to its initial state becomes longer by 98 µm. From the moment of the annealing start (II) the dilatometric sample shrinkage occurs and lasts for app. 800 s. At the finish of the annealing segment (II) (1200 s) the sample length is shorter by 38 µm in relation to the length at the beginning of annealing (Fig. 3).

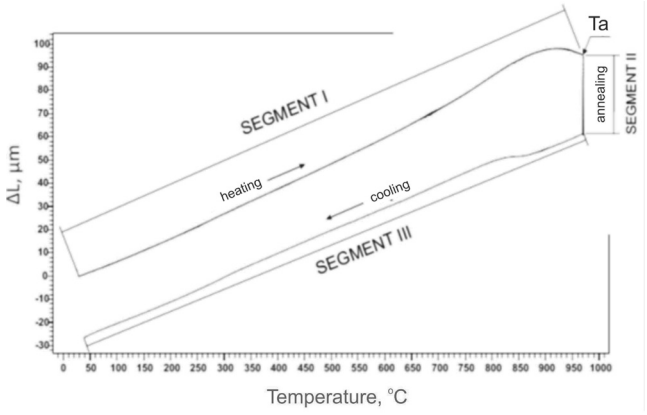

Fig. 2. Scheme of Ti6Al7Nb alloy heat treatment in a system temperature (T) versus elongation (ΔL)

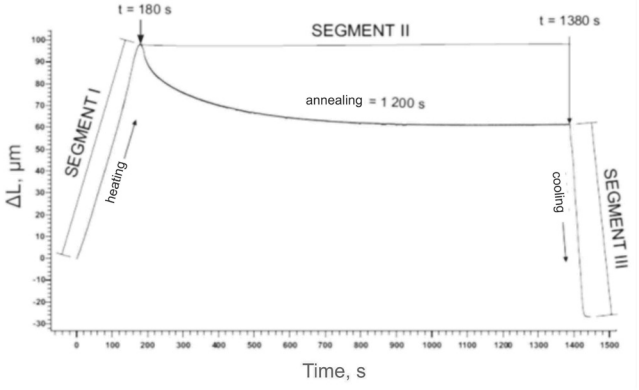

Fig. 3. Scheme of Ti6Al7Nb alloy heat treatment in a system time (t) versus elongation (ΔL)

Microstructures of samples of Ti6Al7Nb alloy obtained after cooling, from a temperature of 970°C to the room temperature with rates: 25; 5; 0.5 and 0.05°C/s, are presented in Figure 4. Observations of Figure 4 indicate that these are bimodal microstructures of quite high dispersion. The application of a temperature lower of app. 40°C than the β–transus temperature is the reason, that a large volume fraction of the bright α phase occurs in the microstructure, since at this temperature it does not

transform into the β phase. The α phase occurs in a nearly globular form-there are large, nearly equiaxial grains. After heating to a temperature of 970°C, annealing and water-cooling, apart from the bright α phase a certain volume fraction of the dark β phase, which at the room temperature is the so-called transformed β phase, is seen in the microstructure. The applied annealing temperature decides on volume fractions of both phases. E.g. applying a temperature of 1000°C, annealing followed by water-cooling causes that the microstructure character is slightly different. This microstructure is presented in Figure 5. Significantly smaller volume fraction of the α phase is clearly visible. As it was already mentioned, after cooling from a temperature of 970°C (with rates 25-0.05°C/s) to a room temperature (Fig. 4) the bright α phase and dark β phase, called transformed β phase occur in the microstructure. It should be noticed that along with decreasing the cooling rate the volume fraction of the dark transformed β phase also decreases. The transformation $\beta \rightarrow \beta_T$ occurs by means of diffusion. Decreasing the cooling rate from 25°C/s to 0.05°C/s causes the volume fraction increase of large, nearly equiaxial grains of the bright α phase. Precipitates of the β_T phase on the α phase grain boundaries, for the slowest cooling rate 0.05°C/s, are of a vermicular character (Fig. 4d).

The detailed results of the hardness of the Ti6Al7Nb alloy samples cooled from 970°C to a room temperature, with various cooling rates, are given in Table 2. As can be seen, the samples hardness is within a narrow range of values: 304-261 of Vickers units. The selected dilatograms of the Ti6Al7Nb alloy cooling from a temperature of 970°C/s to a room temperature with rates: 25°C/s (a); 5°C/s (b); 0.5°C/s (c); and 0.05°C/s (d) together with the corresponding differential curves are presented in Figure 6. The interpretation way of the characteristic temperatures during cooling is shown on dilatograms. Along with decreasing the cooling rate (from 970°C) either a retardation of a length decrease (Fig. 6a-c) or a length increase (Fig. 6d) of the dilatometric sample is observed in the dilatometric curves. This effect is related to a gradual transformation of the β phase into the transformed β phase (β_T) and also to an increase of the α phase volume fraction accompanying the cooling rate decrease. Such explanation of the dilatometric sample length changes and macrostructure changes finds its confirmation in paper [22], where the analogous investigations were performed for the two-phase Ti6Al2Mo2Cr alloy, cooled from a temperature of 870°C (from the two-phase range).

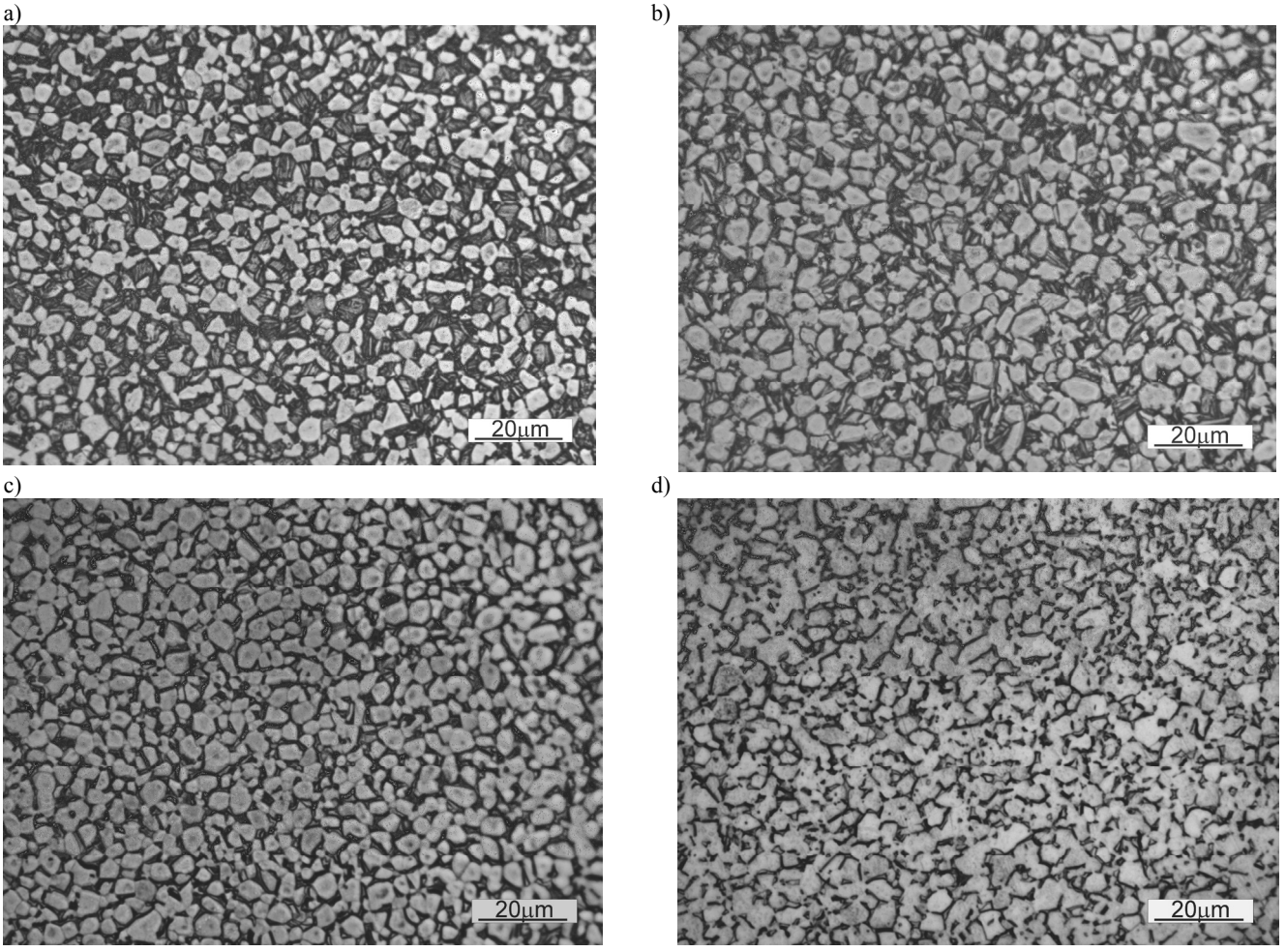

Fig. 4. Optical micrographs of the Ti6Al7Nb alloy cooled from 970°C at: 25°C/s (a), 5°C/s (b), 0.5°C/s (c) and 0.05°C/s (d)

Along with the cooling rate decrease a tendency for the α phase grain growth is seen. It is related to the fact that during cooling the solubility of niobium in the α phase is decreasing. Due to that this element diffuses (it is the easier for niobium the higher was the annealing temperature and the lower cooling rate) to interface boundaries. Spaces from which niobium escaped to interface boundaries became privileged places for the α phase grain growth.

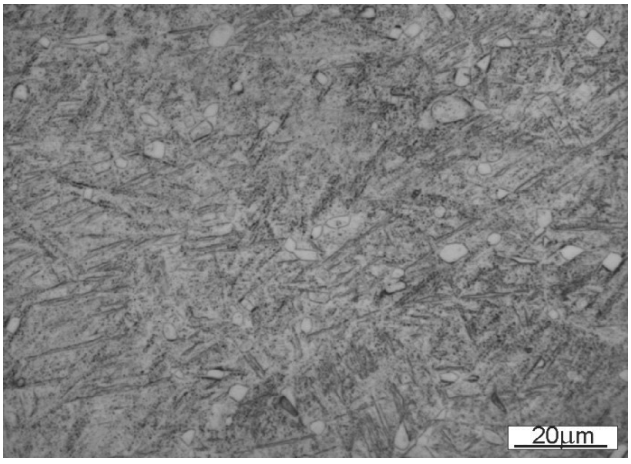

Fig. 5. The microstructure of Ti6Al7Nb alloy after heating to 1000ºC, annealing for 20 minutes and water quenching

The detailed temperature values of the start and finish of the diffusive phase transformation $\alpha+\beta\rightarrow\beta_T$, obtained during cooling the Ti6Al7Nb alloy from a temperature of 970°C with rates: 25÷0.05°C/s, are shown in Table 3. These temperatures are marked as: $(\alpha+\beta\rightarrow\beta_T)_s$ and $(\alpha+\beta\rightarrow\beta_T)_f$.

Along with the cooling rate decrease (within the applied range) the temperature increase of the start and finish of the $\alpha+\beta\rightarrow\beta_T$ phase transformation was seen.

Table 2.
Chemical composition of Ti6Al7Nb alloy according to ISO 5832-11 and heat analysis

Cooling rate, [°C/s]	HV10			$HV10_{med.}$
25	302	306	304	304
5	297	297	294	296
0.5	268	270	266	268
0.05	262	258	263	261

Table 3.
Effect of cooling rate on the detailed temperature values of the start and finish of the diffusive phase transformation $\alpha+\beta\rightarrow\beta_T$ obtained during cooling the Ti6Al7Nb alloy from a temperature 970°C

Cooling rate, [°C/s]	Temperature of phase transformation $\alpha+\beta\rightarrow\beta_T$	
	$(\alpha+\beta\rightarrow\beta_T)_s$	$(\alpha+\beta\rightarrow\beta_T)_f$
25	900	775
5	970	810
0.5	970	840
0.05	970	880

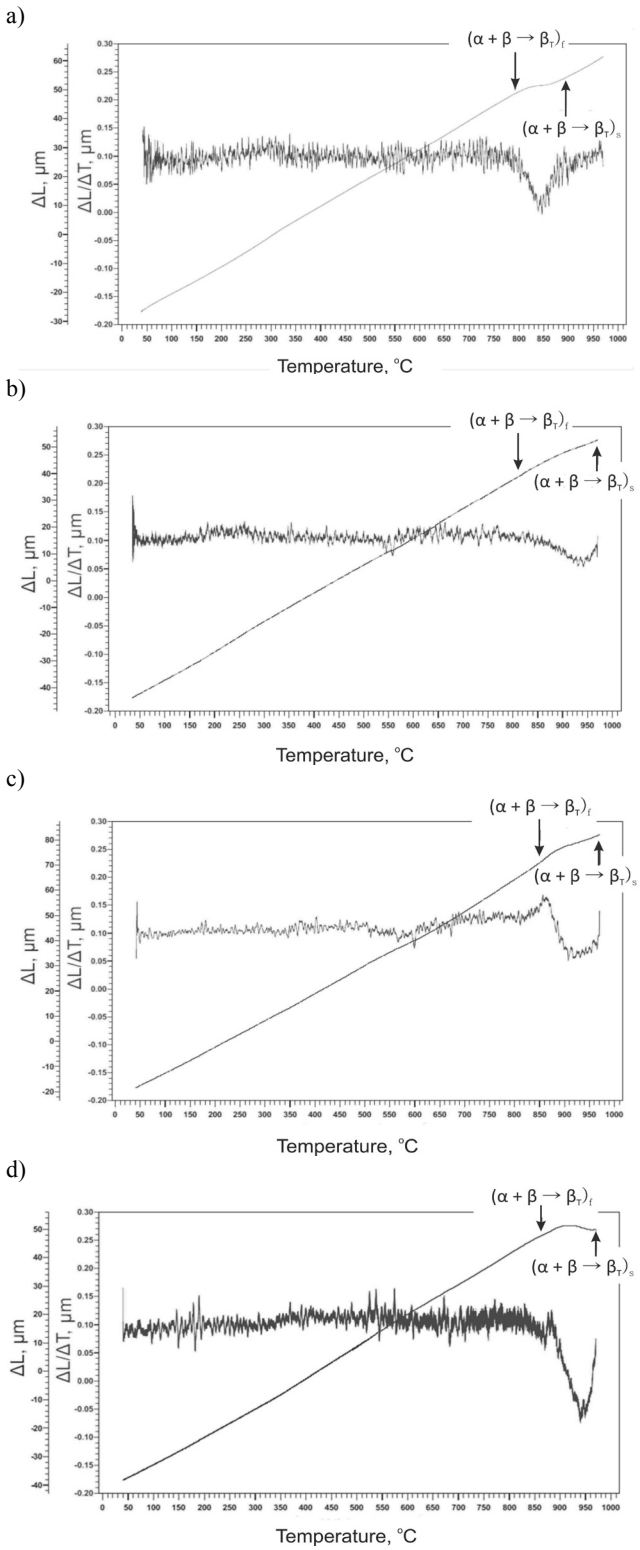

Fig. 6. The dilatometric curves ΔL=f(T) of the Ti6Al7Nb alloy samples cooled from 970°C at the rates: 25°C/s (a), 5°C/s (b), 0.5°C/s (c) and 0.05°C/s (d) and differentiate curves ΔL/ΔT=f(T)

5. Conclusions

The analysis of phase transformations and changes occurring in the microstructure of the two-phase titanium Ti6Al7Nb alloy at a continuous cooling from the two-phase α+β range are carried out in the paper. These analyses are performed for the selected cooling rates, from a range: 25-0.05°C/s, for which the detailed metallographic documentation of microstructures and hardness measurements results are given. The obtained results allow to draw the following conclusions:

1. Along with the alloy cooling rate decrease, in a range from 25°C/s to 0.05°C/s, the transformation of the β phase into the β_T (transformed) phase occurs and the α phase volume fraction increases.
2. A retardation and length increase of the dilatometric samples related to the described above diffusive transformation $\beta \rightarrow \beta_T$ and the α phase grain growth are seen on the dilatometric cooling curves (from 970°C).
3. With the decreased of cooling rate (from 25 to 0.05°C/s) decreased the hardness of the Ti6Al7Nb alloy (from 304 to 261 HV).
4. The obtained results, supported in the future by additional cooling curves, will be used for the development of the original, full CCT diagram of the Ti6Al7Nb alloy cooled continuously from the two-phase α+β range.

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