Georgian Scientists
ქართველი მეცნიერები
Vol. 7 Issue 2, 2025
https://doi.org/10.52340/gs.2025.07.02.10

Determination of the temperature interval of direct and reverse martensitic transformation

in Ti-Nb alloys by means of electrical resistance and differential thermal analysis

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Abstract

In various fields of science and technology, in chemical production, in the fields of food and medicine, the use of binary and multicomponent titanium alloys with different concentrations of chemical elements, which differently affect the phase composition of the alloys and the course of phase transformations, creates the need to study the processes of the formation of metastable phases and structural transformations.

Because of the combined influence of thermal and mechanical processing of the concentration of titanium chemical elements on the nature of phase transformations is poorly investigated, a thorough study of binary titanium alloys with such a β-isomeric element as niobium is required. Its concentration in the Ti-Nb binary alloy determines some features of the martensitic transformation, for example, the tempering-slackening process, which distinguishes it from other alloys. This is the essential importance when comparing the similarities and differences of the mechanism of the metastable phase formation in other titanium alloys during determination of the second component of diffusion mobility in titanium alloys.

The aim of the study was to investigate the shape memory effect in Ti-Nb binary alloys at different Nb concentrations. To determine the superelasticity and damping capacity. To measure the temperatures of the direct and reverse martensitic transformations in the alloy of a given concentration. To determine the reactive tension generated during shape recovery.

Key Words: Titanium, binary alloy, shape memory effect, phase transformation, damping.

The shape memory effect and superelasticity are based on a special type of martensitic transformation - thermoelastic martensitic transformation. [1]. The total energy of such transformation is associated only with thermal and elastic factors. During the transformation, the internal thermal energy decreases, while the elastic, which is caused by the growth of martensite crystals, increases. At a certain temperature, their sum may reach a minimum. In this case, the growth of martensite crystals is stopping and the equilibrium is unstable. This process can be updated by temperature variation or by the action of external forces, which will lead to either further growth of martensite crystals or their disappearance, i.e., a reverse martensitic transformation. The temperature hysteresis of the such transformation is very narrow – in order of tens of degrees. The reversibility of the martensitic transformation in alloys is due to the high degree of compatibility of the crystal lattices of the initial and martensitic phases, and the small amount of the volume mismatches. For a long time, it was believed that the complete reversibility of the martensitic transformation was ensured by the order of the phase lattices. For example, as in the case of titanium nickelide Ti-Ni. However, in the 70s of the 20th centuries, it was proven that the shape memory effect can exist in disordered structures [2]. In the case of an invariant lattice, deformation during the reversible martensitic transformation proceeds by the mechanism of twin formation [3-7]. This type of shape memory effect is characteristic of titanium alloys with the β-isomorphous element Nb [8-17]. In the deformational $\beta \leftrightarrow \alpha"$ transformation, the planes and directions are preserved in both phases, the deformation is close to pure shift deformation. On the tension-deformation curve the shape of the deformational martensitic transformation corresponds to the mechanism of change of the "martensitic flow plateau", the value of which shows the magnitude of the starting tension of the martensitic transformation in the β-phase, which surrounds the martensitic phase. There is no Irretraceable slipp.

At temperatures $T < M_f$ (Completion temperature of the martensitic transformation M_f), alloys contain only the martensitic $\alpha"$-orthorhombic phase. Deformation of alloys at such temperatures leads to the absorption of one binary domain by another [1] as a result of the displacement of the dividing boundary between them. The transformation is realized between those domains whose system is optimally oriented to the acting deformation.

The process can continue until a binary "monodomain" is formed. If the deformation stops before this point, then the number of binary domains will be large. When the alloy is heated to $T > A_f$ (the temperature at which the A_f reverse transformation ends), the so-called reverse recovery occurs. The β-phase does not retain its binary structure. At high voltages, the formation of binary structures is replaced by irreversible slipp. At this point, the degree of shape recovery decreases. Its magnitude depends on the number of binary "monodomains" retained in the alloy.

When $T > M_f$ the alloy contains a cubic volume-centered β-phase. Deformation in this case leads to a deformational $\beta \leftrightarrow \alpha_d''$ martensitic transformation. The athermal $\alpha"$-martensite, which was in the alloy in the initial state of tempering, can change orientation. The shape change caused by the deformational martensitic transformation is maintained after the tension is removed. Heating causes the reverse martensitic transformation and leads to the restoration of the shape.

The shape change caused by the deformational martensitic transformation persists after removing the tension. Heating causes the reverse martensitic transformation and leads to the restoration of the shape.

At $T > A_f$, the β-phase is stable. However, at a certain deformation magnitude, its crystal lattice may undergo a change as in a phase transformation. The resulting martensitic transformation persists only until the removal of the external tension. Removal of the external forces leads to the complete restoration of the stable lattice of the β-phase. The phenomenon of superelasticity is realized. On the tension-deformation curve $\beta \leftrightarrow \alpha_d^{''}$ ($\alpha_d^{''}$ - deformation martensite) transformation is also expressed by a pseudo-flowing plateau.

The crystallographic correspondence $\beta \leftrightarrow \alpha$" during the martensitic transformation is well described by the Burgers relation: $\beta \rightarrow \alpha$ for the martensitic transformation [17, 18]. The magnitudes of the main deformation of the lattice are determined by the relation:

$$\varepsilon_1 = \frac{a}{\alpha_\beta} - 1, \qquad \varepsilon_2 = \frac{b}{\sqrt{2}\alpha_\beta} - 1, \qquad \varepsilon_3 = \frac{c}{\sqrt{2}\alpha_\beta} - 1$$

Here a, b, c are the parameters of the martensite lattice. α_β is the magnitude of the β phase lattice. The magnitudes of the main deformations depend on the concentration of the alloying element.

Increasing the concentration of the alloying element in the alloy reduces the magnitude of the main deformations. They become zero at a concentration above which the β phase becomes completely stable at room temperature. Fig. 1 shows the concentration dependence of the values of the normal modulus of elasticity (E) and the magnitude of the main deformations at the $\beta \rightarrow \alpha$" martensitic transformation (M_s) temperature [19, 20] for a Ti-Nb alloy tempered from 1000°C. The results of phase analysis [19, 21, 22] allow us to conclude that the boundary of the existence of the α" and α" $+ \beta$ regions correspond to the maximum values of the modulus of elasticity (E) (Fig. 1).

It is obvious that the β-phase corresponding to this concentration boundary is very unstable with respect to the $\beta \rightarrow \alpha$" martensitic transformation. It contains a minimum (compared to the concentration of other alloys) amount of alloying element, above which tempering allows us to observe the β-phase. At the same time, the martensitic phase formed from it is maximally supersaturated compared to the equilibrium concentration of the α-phase at the same temperature.

The criteria for the limited instability of the α" and β-phases can be found in [17, 19, 23]. In [19], the limited instability of the α" phase in a Ti-Nb alloy was determined by the dependence of the temperature (M_s) on the concentration of Ta. It was found that α" martensite instability coefficient

$$K = \frac{C_\alpha}{C_{\alpha''}}$$

to be within 4.5÷5.5. Here C_α is the equilibrium concentration of the β-phase at a given temperature M_s, and $C_{\alpha''}$ is the analogous concentration of α" martensite at the same temperature.

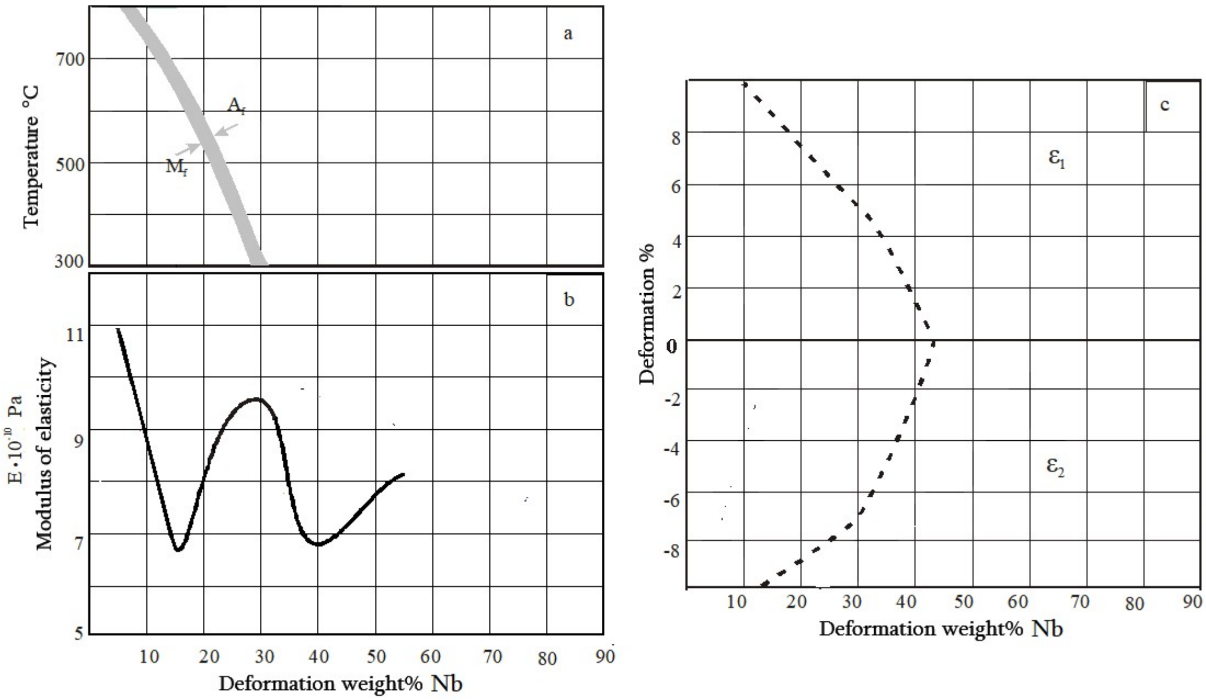

Fig. 1. Concentration dependence of the normal modulus of elasticity E and ε_1, ε_2 values of the martensitic transformation temperature (M_S) of Ti-Nb alloy.

The estimation of the limited instability of the β-phase is given in [17, 18]. Instability coefficient

$$K_\beta = \frac{C_\beta}{C_\beta^{lim}}$$

Here C_β is the concentration of the alloying element in the given alloy, C_β^{lim} is the concentration in the limited unstable β-phase. It is obvious that in the case of limited unstable alloy $K_\beta = 1$.

Experiments show that Ti-Nb alloys for which K_β is ~(0,9÷1.2) has practical importance. As can be seen from Fig. 2, Ti-Nb alloys for which K_β ~ 25.9÷33.1 wt%Nb are within this range.

Ti-Nb alloys with Nb contents of 25.9, 29.8, 31.1 wt% were obtained by electron beam melting. Due to the technical limitations of the electron beam melting equipment available to us, the mass of a single alloy did not exceed 50 g. The melting took place under vacuum conditions of 10^{-4} mm of mercury column. To achieve maximum uniformity of alloy, the blocks were melted 5 times and then homogeneously burning at 1000 °C for 50 hours. The blocks were rapping and by hot rolling method the plates of 3÷5 mm thickness was produced.

Table 1 shows the percentage composition of the investigated Ti-Nb binary alloy. For further thermomechanical testing and to investigate the physical properties of the alloys, the alloys were cut into flat parallel samples with dimensions of 5×10×100 mm and 3×10×100 mm. Cutting was performed on an electric erosion machine, which eliminated the formation of mechanical tensions in the samples. Subsequently, grinding was carried out with an abrasive disc under water cooling.

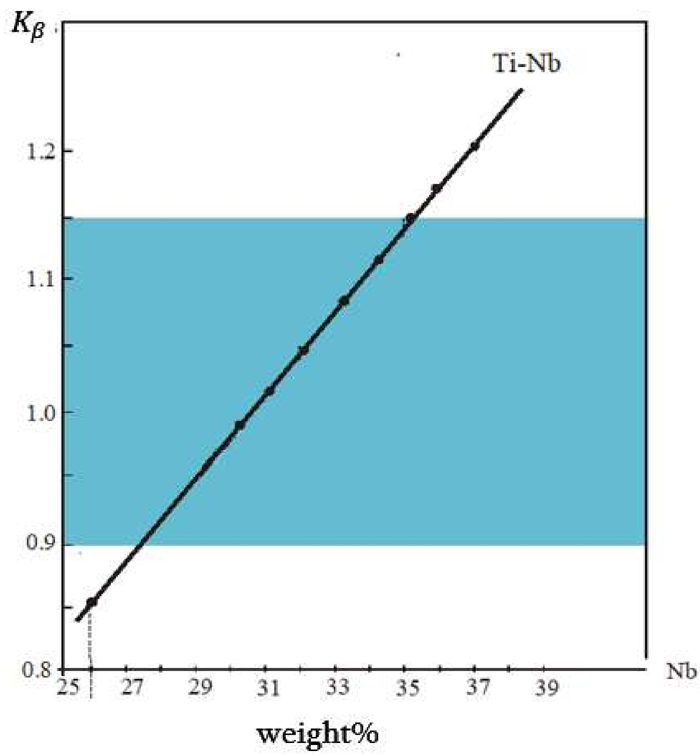

Fig. 2. K_β dependence on Nb concentration in binary titanium alloys.

Due to defects introduced during grinding, the samples were homogenized at 800-900 °C for 0.5-1 h. For X-ray structural examination and metallography, the surface layer was poisoned with a standard solution for titanium alloys: one part of a 40% solution of HF and three parts of HNO3 (density 1.4 g/cm^3).

The samples of alloy were thermaly treated at various temperatures by tempering to obtain a phase composition in which the deformational martensitic transformation is most complete.

The X-ray structural analysis determined the type and sequence of phase transformations in the alloys.

Table 1

Component content in % by weight

Ti	Nb	Zr	O	N	H
other	25.9		0.15 – 0.20		
	29.8				
	33.1				
	33.0	7.1			
	26.08	6.95			

Depending on the niobium concentration, three types of microstructures were observed: α" orthorhombic metastable martensite, with a small amount of $\alpha" + (\beta)$ austenite, and a mixed structure $\alpha" + \beta$. The microstructure had a significant impact on the deformation process of the alloys. The results are presented in Table 2. In order to reduce the phase transformation temperature, we added a certain amount of Zr to the binary alloys, which significantly reduced the martensitic transformation temperature and had a positive effect on the shape memory effect.

Table 2

Dependence of phase composition on component constituent and concentration of the components.

Ti-25.9 weight% Nb α″	Ti-29.8 weight % Nb α″+(β)	Ti-33 .1weight % Nb α″+β
Ti- 33.0 Nb - 7.1 Zr α″+β	Ti-26.08 Nb - 6.95 Zr α″+β	

Existence of a thermoelastic $\beta \leftrightarrow \alpha"$ transformation in titanium alloys is confirmed by analyzing of the temperature dependence curves of differential thermal and electrical resistance.

A series of differential thermal analysis and temperature dependence studies of electro resistance was carried out for determination of initial and final temperatures of thermoelastic direct and reverse martensitic transformations in alloys tempering from 1000°C. Pure titanium was used as the standard for differential thermal analysis. The results of such studies for Ti-Nb binary alloys are presented on Fig. 3.

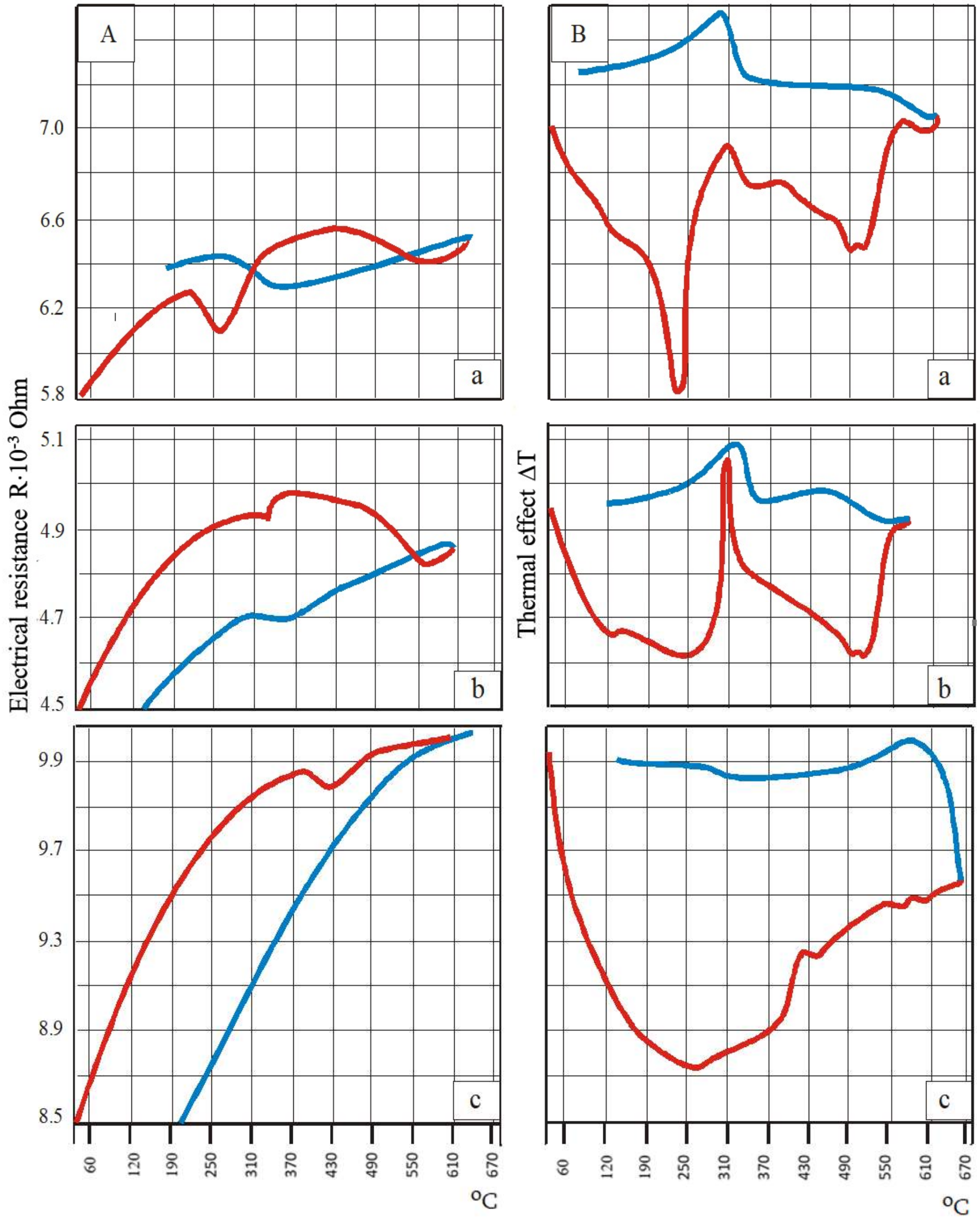

Fig. 3. Temperature dependence of electrical resistivity (A) and differential thermal analysis (B) curves for alloys: Ti-33.1 wt% Nb (a); Ti-29.8 wt% Nb (b); Ti-25.9 wt% Nb (c). (Heating - cooling).

They indicate the complex nature of structural changes in alloys. These changes can have a significant impact on the non-elastic properties of alloys. The main regularities of the transformations are observed for all investigated binary alloys (Table 3).

Table 3

Dependence of phase transition modulus and martensitic transformation temperature on Nb concentration in binary alloys.

	Concentration of alloing elements in wt%	Phase composition	Modulus of elasticity	Martensitic transformation temperature range (t°C)	
	Nb			$M_s - M_f$	$A_s - A_f$
1	25.9	α''	9,24	375-345	365-395
2	29.8	$\alpha''+(\beta)$	9,87	285-255	280-320
3	33.1	$\alpha''+\beta$	7,83	205-170	190-250

The table clearly shows the regularities of the processes occurring during heating and cooling in alloyed alloys. For maximal exposing of the effects of experimentally selected thermomechanical treating these curves (Fig. 3) can be used as indicators of the structure of the alloys.

The influence of Nb concentration on the course of phase transformations in binary alloys is clearly seen in Fig. 3. Table 3 shows the dependence of the phase composition on concentration determined by the X-ray analysis method. It is obvious that with increasing Nb concentration, the martensitic transformation temperature decreases and a second, "high-temperature" endothermal effect appears next to the "low-temperature" endothermal effect, while the magnitude of the exothermic effect increases on the cooling curve (Fig. 4).

Also, the course of transformations is well expressed on the curve of the temperature dependence of the electrical resistance for binary alloys.

An increase in temperature in the regions of phase transformations causes a sharp change in the sign of the temperature coefficient of resistance on the resistance curve.

The results of our temperature dependence of electroresistance and differential thermal analysis were compared with the results of a French-made computerized calorimeter. Since the containers used in the calorimeter were made of aluminum (melting point 630°C), a certain amount of zirconium was added to the Ti-Nb alloys to reduce temperatures of phase transformation for fixing the high-temperature phase transformation. The graph of the heating-cooling cycle of the calorimeter study is shown in Fig. 5.

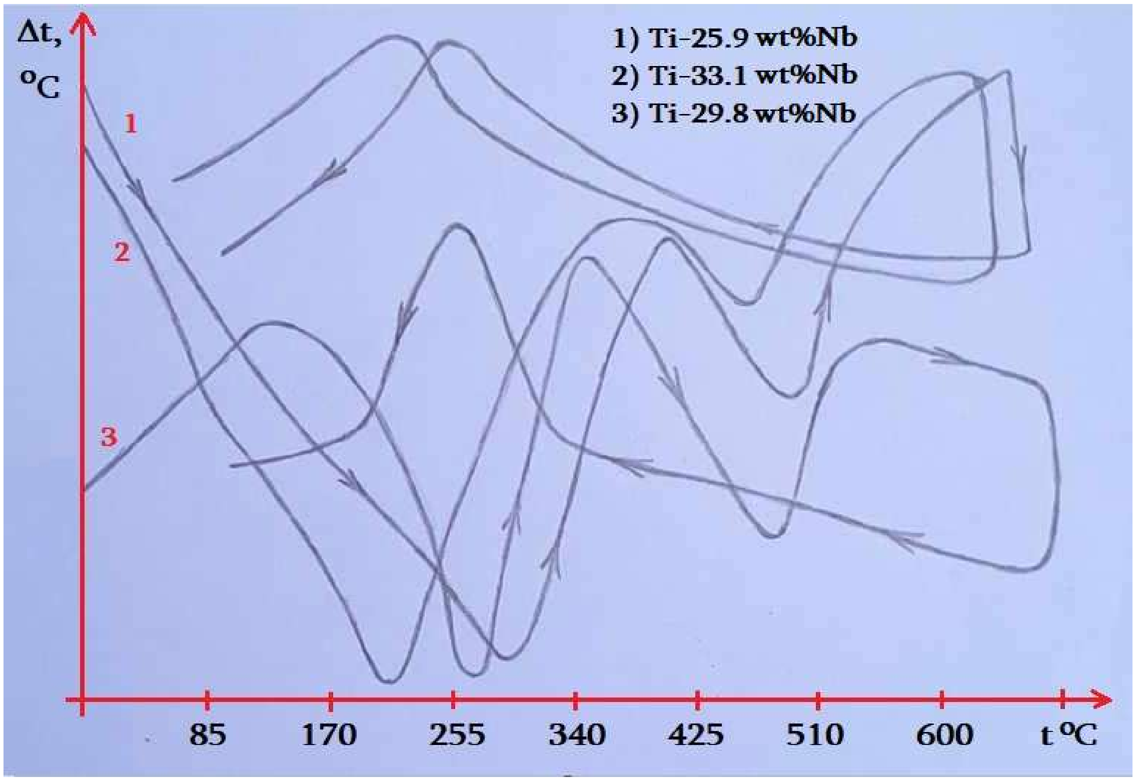

Fig. 4. Dependence of the temperature of phase transformation on the concentration of niobium in the alloy

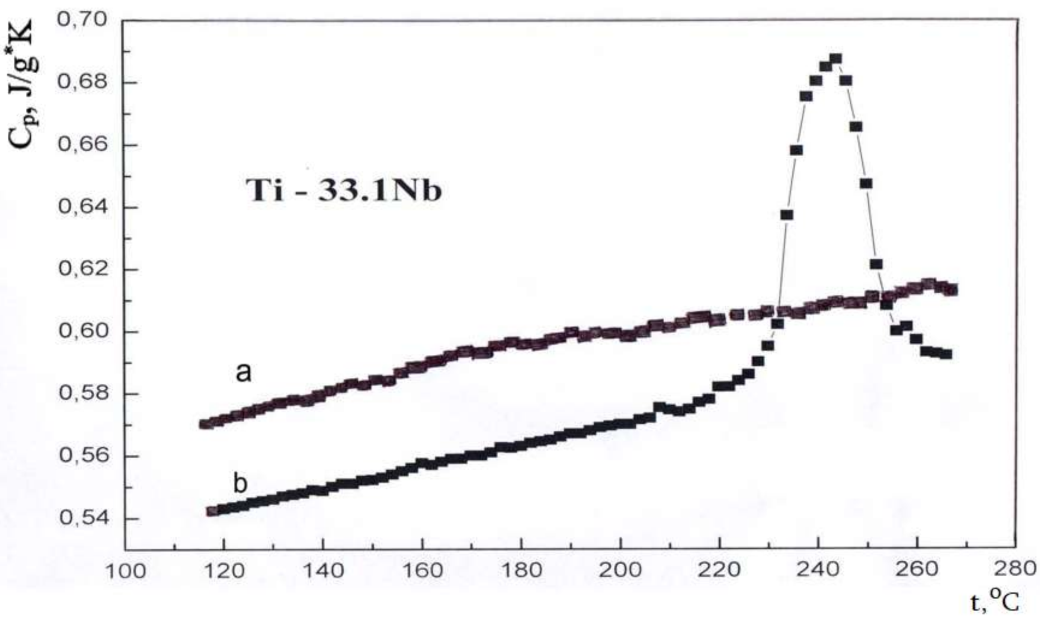

Fig. 5. Calorimetric curve of the first ("low-temperature") endoeffect demonstration.

Conclusion

Literature data on the supercelasticity and damping properties of Ti-Nb shape memory binary alloys based on titanium were analyzed.

Special attention was paid to the study of the non-elastic properties of Ti-Nb binary alloys and factors for improving their detection. Based on the analysis of literature data, the percentage composition of Ti-Nb binary alloys was selected for observation.

Binary and Zr-doped alloys with optimal K_β of Ti-Nb were obtained. To achieve the required equilibrium state, a technology of thermomechanical processing of the alloys was developed.

X-ray structural and calorimetric studies of the alloys were carried out, and the temperature dependences of the differential thermal and electrical resistivity of the phase transformations occurred were analyzed.

The macroscopic detection of phase transformations resulted in two effects of shape memory observed in the investigated alloys during mechanical loading: relatively low-temperature martensitic and high-temperature diffusional. Accordingly, when the alloys were heated after deformation, quite large reactive tensions were induced in them.

The influence of thermomechanical processing on the appearance of the shape memory effect and reversibility was studied.

During investigation, the influence of the heating-cooling speed on the degree of the shape memory occurance effect was revealed. In many cases, the heating speed of the sample, together with the mechanical load, played a decisive role in the course of phase transformations and appearance of the shape memory effects associated with it.

Based on the analysis of phase transformations and shape memory effects, all alloys showed two shape memory effects.

From the analysis of the results, we can conclude that the obtained binary alloys are promising materials in terms of their use in aerospace products. Also, the corrosion resistant and biological inertness of the investigated materials indicate the prospects for their use in various fields of medicine, chemical, food industry and technology.

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Ti-Nb-ის ბინარულ შენადნებში პირდაპირი და შებრუნებული მარტენსიტული გარდაქმნის ტემპერატურული ინტერვალის დადგენა ელექტრო წინაღობისა და დიფერენციალური თერმული ანალიზის საშუალებით

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აბსტრაქტი

მეცნიერებისა და ტექნიკის სხვადასხვა დარგებში, ქიმიურ წარმოებაში, კვებისა და მედიცინის სფეროებში ტიტანის ბინარული და მრავალკომპონენტიანი შენადნების გამოყენება სხვადასხვა კონცენტრაციის ქიმიურ ელემენტებთან, რომლებიც განსხვავებულად მოქმედებენ შენადნების ფაზურ შემადგენლობაზე და ფაზური გარდაქმნების მსვლელობაზე, ქმნის მეტასტაბილური ფაზების წარმოქმნის და სტრუქტურული გარდაქმნების პროცესების შესწავლის აუცილებლობას.

რადგან ტიტანის ქიმიური ელემენტების კონცენტრაციის თერმული და მექანიკური დამუშავების ერთობლივი გავლენა ფაზური გარდაქმნების ხასიათზე ნაკლებად არის გამოკვლეული, საფუძვლიან შესწავლას მოითხოვს ტიტანის ბინარული შენადნობები ისეთი β-იზომერულ ელემენტთან, როგორიცაა ნიობიუმი. Ti-Nb-ის ბინარულ შენადნში მისი კონცენტრაცია განაპირობებს მარტენსიტული გარდაქმნის ზოგიერთ თავისებურებას, მაგალითად წრთობა-მოშვების პროცესი, რითაც ის განსხვავდება სხვა შენადნებისაგან. ამას არსებითი მნიშვნელობა აქვს ტიტანის სხვა შენადნებში მეტასტაბილური ფაზის წარმოქმნის მექანიზმის მსგავსებისა და განსხვავების შდარებისას ტიტანის შენადნებში მეორე კომპონენტის დიფუზიური ძვრადობის განსაზღვრის დროს.

კვლევის მიზანი იყო Ti-Nb ბინარულ შენადნებში Nb-ის სხვადასხვა კონცენტრაციის შემთხვევაში ფორმის მახსოვრობის ეფექტის გმოკვლევა. ზედრეკადობის და დემპირების უნარის დადგენა. პირდაპირი და შებრუნებული მარტენსიტული გარდაქმნების ტემპერატურების გაზომვა მოცემული კონცენტრაციის შენადნში. ფორმის აღდგენის დროს წარმოქმნილი რეაქტიული ძაბვების განსაზღვრა.

საკვანძო სიტყვები: ტიტანი, ბინარული შენადნობი, ფორმის მახსოვრობის ეფექტი, ფაზური გარდაქმნა, დემპფირება.