Production and Application Features of Implants from Nitinol with the Shape Memory Effect

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INTRODUCTION
The paper describes the outcomes of a research of a mechanical behavior of implant - staples from TN1 alloy, used for fixing of cutted breast bone during cardiological operations. The connection of breast bone is carried out by one-way shape memory effect. The influence of various characteristics, such as test temperature, degree of strain during the loading, and cycle load, on a mechanical behavior of staples have been considered.

Key words: nitinol, shape memory effect, implant.

The alloys on the base of nitinol (Ti-54÷57 % per mass) have high corrosion resistance in many aggressive environments and biological compatibility with tissue of man's organism [1]. Their mechanical behavior under loading is determined by temperature, and they can be carried out to similar behavior of rigid tissues of a man due to special heat treatment. These alloys have a special functional properties, such as shape memory effect and superelasticity [2]. Combination of those properties has defined high interest to alloys on the base of nitinol as to materials for implants and medical tools. At present several dozen of different staples, used for connection of bones and another kind of operations, are developed [3]. However, the application of this material in medicine is limited. This fact is explained by difficulties in manufacturing of semifinished products from this alloy, and because the technology of application and serviceability of products are investigated unsufficiently.
The most important properties of alloys on the base of nitiol are the start and finish temperatures of shape recovering. For the products used in medicine, these temperatures are strictly limited and should varied in interval 25÷45°C. The temperatures of direct and inverse martencite transformation are depend on chemical composition and structural condition of alloy. Therefore, the optimal composition of alloy and technology of following treatment will provide required level of service characteristic reliability.
The application of Ti-Ni alloys annealing at 700-900°C allows considerably change the characteristics of shape memory effect and shape recovering temperatures. This change connected with dissolution of intermetallic compounds such as Ti_3Ni_4, Ti_2Ni_3 in B2-phase. Inverse extraction of intermetallics not happens during cooling from annealing temperatures. The composition of B2-phase differs from equilibrium composition. Aged can leads to equilibrium structure of alloy. Thus the B2-phase composition should change and, as a consequence, the shape recovering temperatures should change too. The researches of influence of chemical composition of alloys on the base of nitinol and heat treatment to range of changing characteristics such as shape memory effect and superelasticity [2, 4]. Dependence of limits of changing start and finish shape recovering temperatures (A_s^1, A_f^1) from chemical composition is showed on Fig.1. The minimal level of shape recovering temperatures has been received by annealing of alloys at 700-900°C, and the maximal lever - by ageing at 450°C.
According to the outcomes of the researches the alloy TN1 (Ni-54,8÷55,8 % per mass.) was selected for production of implants. The composition of this alloy allows to change the shape recovering temperatures from 25°C to 45°C by thermal and thermomechanical treatments, that's corresponded to medical requirements. Semifinished products were exposed to cold or hot deformation and annealing at 450-600°C for giving required shape to finished product. Final thermal treatment is carried out at 450-550°C, that is provided shape recovering temperatures. Temperature and time of annealing are varied. Therefore the structural condition of B2 - phase (polygonized, recristallized) and content of Ni are changed due to dissolution and extraction of intermetallics, such as Ti_3Ni_4 and Ti_2Ni_3.

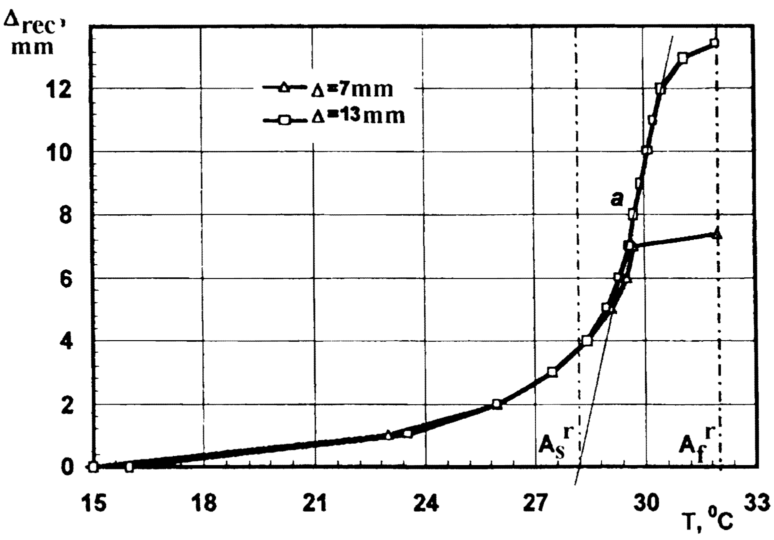

Fig. 3. Recovering of staple shape (Δ_{rec}) deformed at 10°C on 7 and 13 mm.

The Fig. 3 shows that the implant shape recovering curve can be devided on two parts. At the first stage the process of shape recovering develops slowly with the temperature increase. The rate of shape recovering sharply increase during staple heating above some temperature. To determine this temperature it is enough to put tangent line through the point *a* up to intersection with an axes of abscissas. The implant heating up to this temperature allows recovering not more then 10-20% from attached deformation. That's why this temperature is the start recovering temperature (A_s^r). Temperature A_s^r of researched staple lied in interval 26÷29°C, that is quite satisfies the required condition. Fig. 3 shows that the shape recovering kinetics does not depend on a degree of strain. It's necessary to mention that this statement is correct only if the staple deformation does not exceed critical value, after which full shape recovering during heating does not happen [5].

The researched staples are installed to the loading mechanism and heated in interval 8÷90°C (accuracy 0,1°C) for determination their serviceability characteristics.

Schemes of tests:

1. Deformation (load - unload) on different value (Δ) at constant temperature.
2. Deformation on fixing value (Δ=const) and heating up to prescribed temperature with the rate 0,3°C/s.
3. Deformation on fixing value, unloading and heating with rate 0,3°C/s in grippers of loading mechanism with rigidity 10 N/mm.

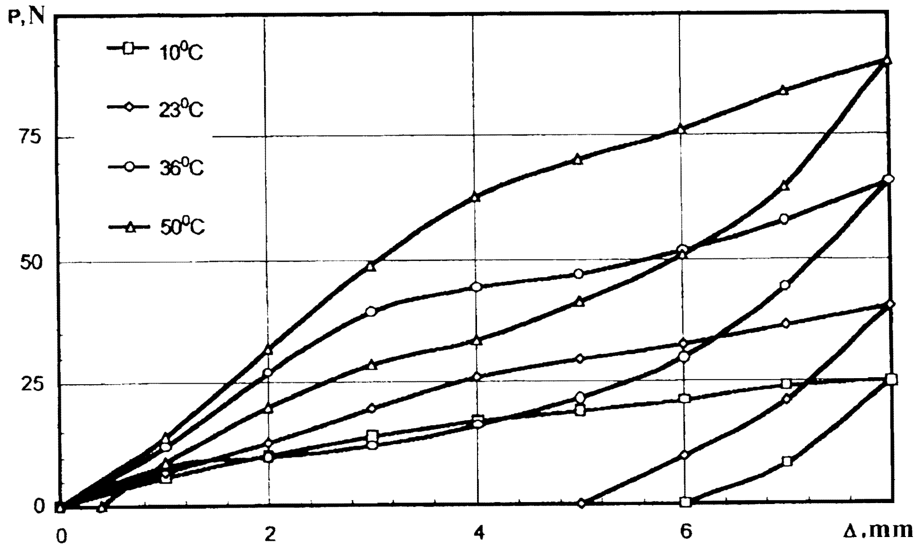

Fig. 4. Influence of temperature to mechanical behaviour staples from alloy TN1.

Deformation curves, received during tests under scheme 1 (Δ=8 mm) at the different temperatures, are showed on Fig.4.The staple has low plastic strain efforts and high residual elongation under load at 10°C. Such mechanical behaviour is usual for material with temperature of inverse martencite transformation (A_s), which is lower then test temperature. In this case the formation of martencite, oriented in the corresponding with strains, results from loading and exists in alloy

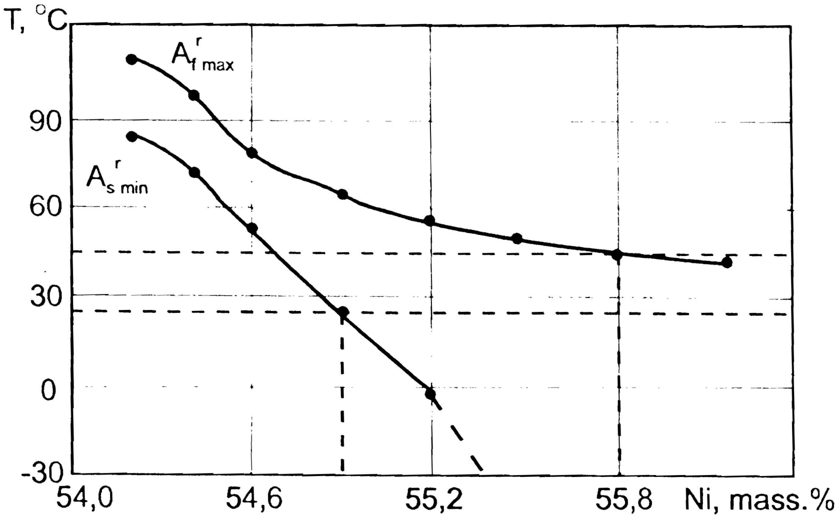

Fig. 1. The influence of Ni content to A_s^r (annealing at 700-900°C) and A_f^r (annealing at 450°C).

Researchers of a mechanical behaviour of staples, used for fixing of cutted breast bone during cardiological operation (Fig. 2), made from an alloy Ti-55,3% Ni are indicated below.

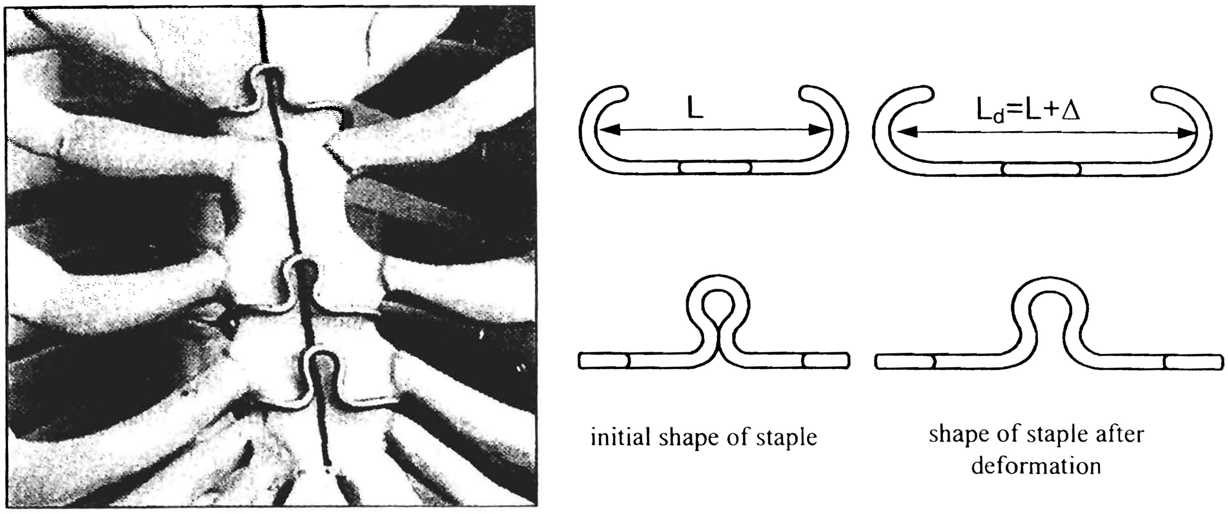

Fig. 2. Scheme of cardiological staples installation.

Connection of cutted bone by staple is carried out by realization unilateral shape memory effect. The principle of shape memory effect is the shape recovering by heating after accumulation the strain in material with stress martencite structure.The working temperatures of implants is the temperature of man's body. That's why the temperature of finish inverse martencite transformation (A_f) shouldn't exceed 35÷36°C. This condition has been provide due to optimal regime of semifinished product thermal treatment.

During the operation the staples are deformed at temperature below 19°C, so that increase the size L (Fig.2) on Δ=6÷12 mm (Δ=L_d-L, L and L_d -initial staple size and staple size under deformation). Deformed staple is installed on connected breast bone, which size is the bigger then the initial size L on 4÷7 mm. After heating above 27°C the staple has a tendency to recovering initial shape and at 36°C the staples constricts the breast bone.

Air temperature in operational room is supported by a constant and equal 23°C. Therefore, in order that the deformed staple will not recovered the shape prematurely during the operation, the temperature of start of shape recovering of implant shouldn't be below 23°C. Fig. 3 shows the dependence of shape recovering from temperature (Δ_{rec}=L_d-L_t, L_t - staple size at t°C).

after unloading [2]. The shape changing of material is explained by only martencite transformation mechanism and twinning effect. Besides, there is no sliding completely, that is confirmed by full recovering of initial shape of deformed staple during the heating in free condition above 35°C.

Increase of plastic strain efforts and decrease of residual strain during loading (Fig. 4) are observed in the time of staple deformation in interval of inverse martencite transformation (A_s=27°C; A_f=35°C). The inflection of a stress curve separates the linear site of dependence efforts from Δ from more slanting site. Moreover the efforts values, corresponded to inflection of a curve, increase with temperature growth. That can be explained by increase of strains of martencite formation during loading and it partial or full transition in initial phase during unloading.

Material demonstrates a characteristic superelastic behaviour at deformation temperature above A_f. In this case, during exceeding of some critical degree of strain, full returning to initial shape is not observed after unloading or additional heating. It is called by that the martencite transformation stresses are so great during loading, that they approach to slide stresses. Sliding developments calls mechanical and thermal irreversible shape changing of staples [5]. And the more higher test temperature, the shape changing is carried out by sliding mechanism in the greater degree, and the degree of residual non-recovering strain of staples is higher.

As the service temperature of implants is about 36,6°C, we'll consider mechanical behaviour of staple at that temperature in detail (Fig. 5). Stress curve of staples has two brightly expressed inflections:

- the first inflection (during deformation on 3 mm) is caused by start of stress martencite formation;
- the second inflection (during deformation on 10 mm) is caused by start of shape changing by sliding mechanism.

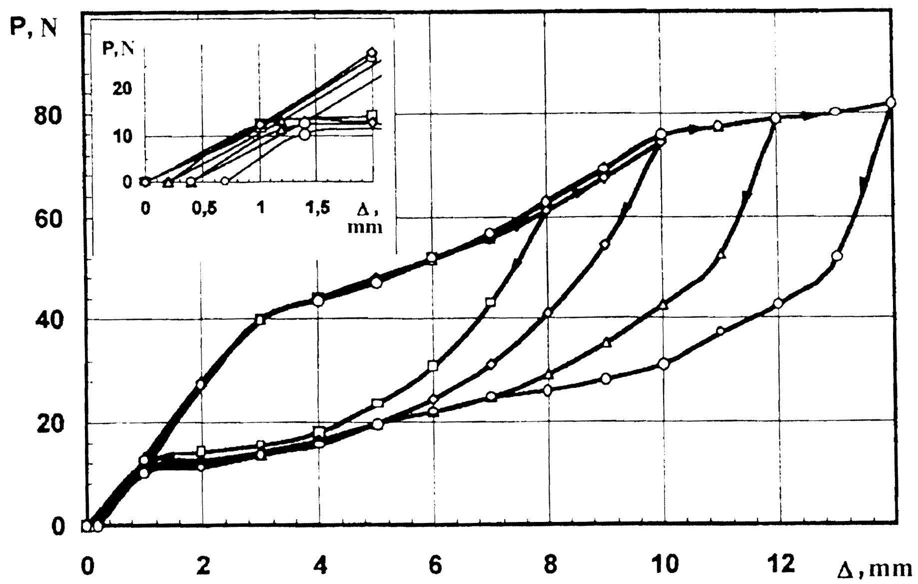

Fig. 5. Deformation curves of implants (staples) from alloy TN1 at 36,6°C.

Unloading of staple, deformed up to 10 mm, lead to full realization effect of superelasticity, and the accumulation of residual thermal irreversible strain happens during large strain. The greatest degree of strain develops in its loop. Degree of strain of staple loop, over which the sliding develops, equal approximately 12% at 36,6°C, that is well corresponded with results of research [5]. This paper determines the maximal possible superelastic strain without sliding of alloy TN1.

Staple should develop a force during installation to the patient. The staple size L should be less then size of breast bone for providing required force. The staples were deformed at 10°C on Δ=9÷14 mm and heated up to 36,6°C in the loading mechanism for determination required force. Besides the grippers of loading mechanism are fixed so that the Δ were equal 4÷7 mm at 36,6°C. The forces, developed by staple during the heating, correspond with forces, developed by implant under unloading in the superelastic condition at the same temperature (Fig. 6). The value of force and Δ (in which the equilibrium between implant and elastic elements of the loading mechanism are set up at fixed temperatures) depend on rigidity of loading mechanism. Rigidity of loading mechanism is determined by a tangent of declination angle of section 3-4 (3'-4') (Fig. 6). Results of implants tests under the schemes 1-2-3-4, 1-2'-3'-4' and 1-2'-3'-3-4 (Fig. 6) show that the force, developed by staple, depends on Δ and does not depends on a degree of preliminary strain.

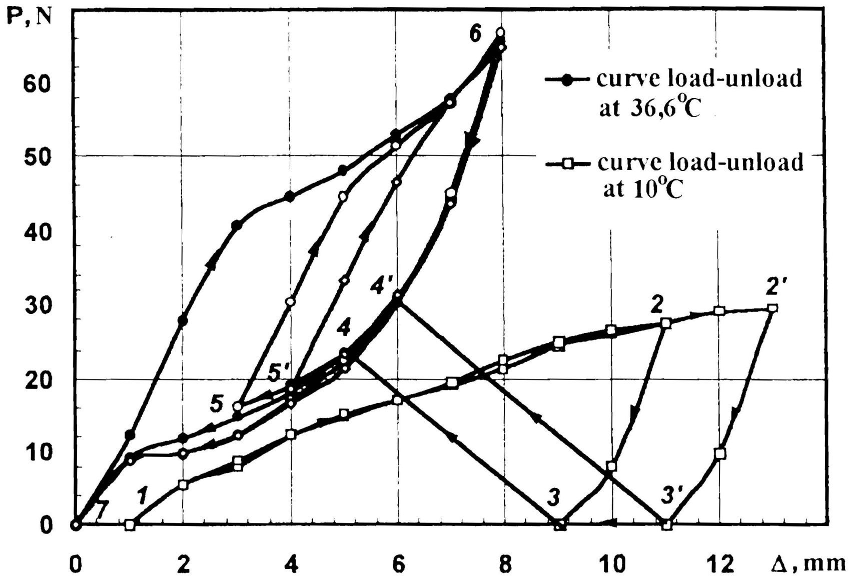

Fig. 6. Mechanical behavior of implants (staples) during changing of deformation and test temperature.

After the condition of equilibrium was achieved, the changing of equal Δ under scheme 4-5-6-7 (4'-5'-6-7) has been accompanied with effort changes. Developing forces are changed along unloading curve in superelastic condition during decreasing of Δ. And during increasing - the forces tend to appropriate values on loading curve. After installation the staple to the patient, during knitting consolidation of breast bone (up to 6 months), it should provide a rather smooth changing of load during the cycle deformation (during breathing, cough, physical exercises). The excessive rigidity of staple can result to facing of bone during stress and inadmissible decrease of developed forces during unloading.

The staples were deformed under the schemes 2 and 3 for imitation of service conditions so that the Δ equaled 5 mm after heating up to 36,6°C. After that the staple cyclically deformed (up to 10 cycles) relatively determined Δ on 1,2 and 3 mm. The tests have shown (Fig. 7) that the staple mechanical behaviour stabilize on the closed loop and not depend on tests scheme after 2÷3 cycle of loading-unloading. The loop width increase during increase of cyclical deformation, and the maximal and minimal efforts in cycle approach to corresponding value on loading-unloading curves. Average rigidity of staple equal approximately 12 N/mm in the time of cyclical deformation.

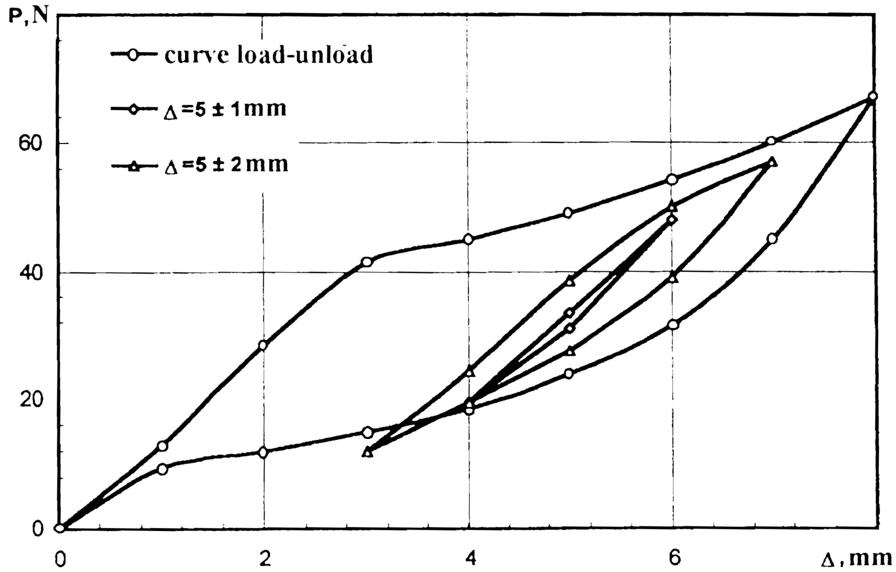

Fig. 7. Mechanical behavior of implants (staples) from alloy TN1 during cyclical deformation at 36,6°C.

The changing of start and finish temperatures of martencite transformation by heat treatment of semifinished products can be used as a method of management serviceability characteristics (Fig. 8). The level of forces developed by staple at 36,6°C, increases during the decrease of inverse martencite transformation temperatures and during the increase - decreases. It's necessary to mark, that in case when the temperatures of finish of shape recovering exceeds 36,6°C, application of warm physiological solution is required for full shape recovering of implant during operation. Besides, the staple partially loses the superelastic properties under cyclical deformation and at the same temperature. This fact limits a possibility of application such implants in practice.

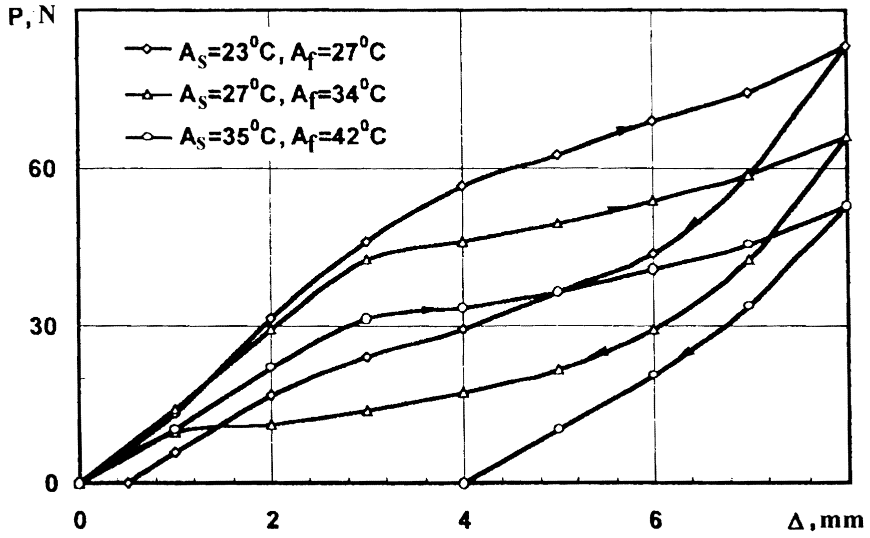

Fig. 8. Mechanical behaviour of implants (staples) with different A_s and A_f at 36,6°C.

Fig. 8 shows that the staple with A_f=42°C didn't recovered the shape up to extremity after deformation on Δ=8 mm and following unloading at 36,6°C. The staple requires additional heating for full shape recovering. That can be explained by partial inverse martencite transformation. The partial shape recovering is observed in staple with A_f=27°C after load-unload process at the same conditions. The reason of this fact is a sliding in material after staple deformation on Δ=8 mm. This process leads to thermally irreversible shape changing.
The forces, developed by staple at 36,6°C, can be increased by additional overheating above prescribed temperature (Fig. 9, 10).

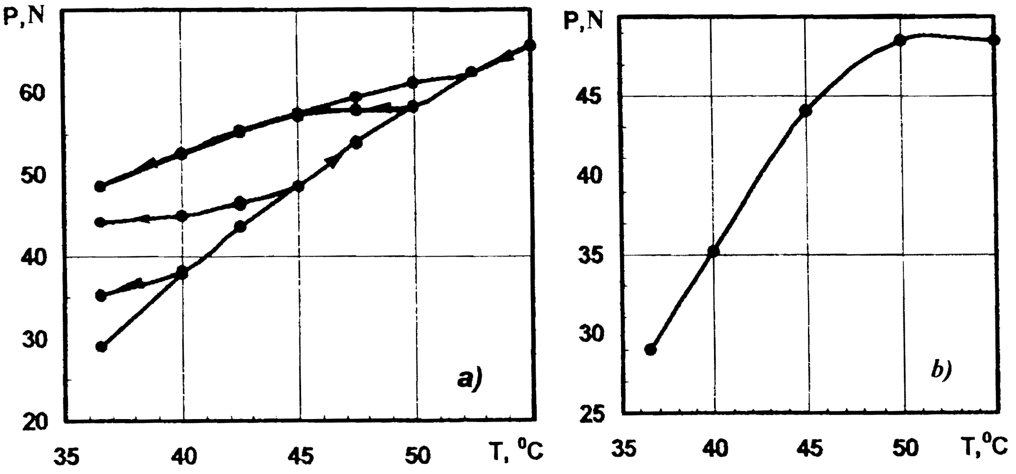

Fig. 9. Influence of overheating temperature to the force , developed by implant A_s=27°C, A_f=34°C, Δ=7 mm:
a) changing of developed forces during heating and cooling of implant;
b) dependence of developed forces at 36,6°C from overheating temperature.

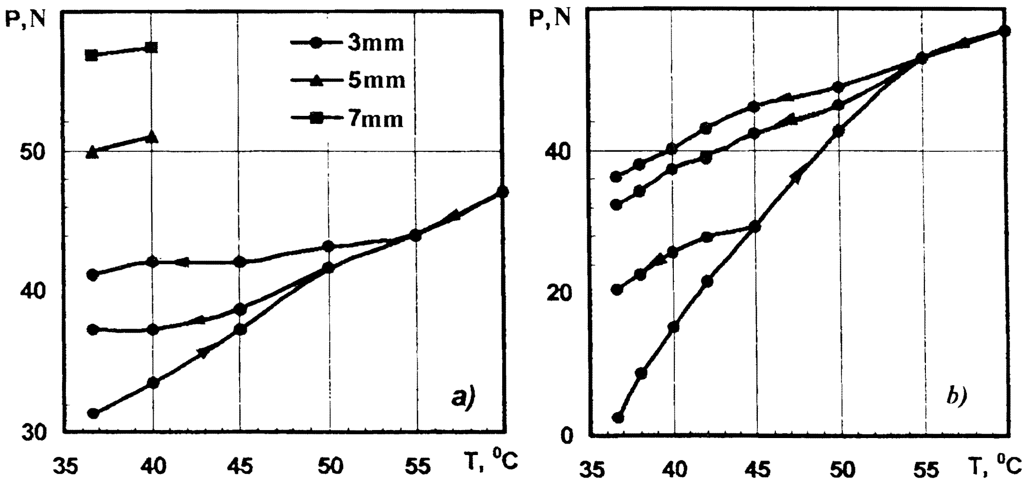

Fig. 10. Influence of overheating temperature to the force, developed by implant:
a) A_s=23°C, A_f=27°C;
b) A_s=35°C, A_f=42°C.

The staple were deformed at 10°C and heated up to 36,6°C for evaluation of influence of overheating to force, developed by implant. The grippers of loading mechanism are fixed so that the Δ were equals 7 mm at 36,6°C. The Δ=const during the following staples heating. The heating is carried up to 40, 45, 50 and 55°C. The force, developed by staple, increases after overheating above 36,6°C and following cooling to former temperature (Fig. 9). This force increases up to some limited value in the time of increase of overheating temperature (Fig. 9b).Overheating of implant above temperature, corresponding of maximal value of force, can leads to sliding process in material. Therefore the overheating temperature of implants with A_s=27°C and A_f=34°C and Δ=7 mm shouldn't exceed 50°C.

Thus researchers allow to formulate principle of application of staples from alloys on the base of nitinol with the shape memory effect. At the first, the initial staple size should be less then breast bone width on 5-7 mm. In this case the deformation, developed in staple after heating up to 36,6°C, not exceed 10% and provide required force of constriction.

The exceeding of prescribed difference of staple size and installation place can lead to exceeding critical level of deformation and irreversible staple shape changing during service by developing of sliding process. The decrease of difference below 5 mm does not allow to provide the developing of force for connection of breast bone during heating up to 36,6°C.

At the second, the decrease of inverse martencite transformation by heat treatment can leads to increase the level of force, developed by staple at 36,6 °C. So for implant with A_s=23°C and A_f=27°C the forces increases no less then 25% on a comparison of implant with A_s=27°C and A_f=34°C. Therefore, it's necessary to take into account, that the increase of difference between inverse martencite transformation temperature and service temperature leads to developing of sliding process in material due to decrease of value of critical deformation. It is necessary to reduce a difference between staple size L and size of installation place.

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