Cyclic deformation of Ti-Ni alloy used for endovascular device

T.V. Morozova[1*]

[1]National University of Science and Technology "MISIS", Moscow 119049, Russia

Abstract. Has been studied the possibility of using TiNi alloys with shape memory and pseudoelasticity as an implantation material for correction of functional mitral regurgitation endovascular device. The modes the deformation cycling were developed that allows fulfilness of necessary properties eligible to operate power-unit device for trans-skin implantation method. Preliminary deformation cycling provides mechanical properties range stabilization that guarantees the shape reinstatement of implant after its installation.

1 Introduction

Shape memory and pseudoelasticity alloys have good biocompatibility and corrosion resistance, which make them ideal for biomedical applications. Superelastic NiTi is the optimal material for medical applications, as it allows to overcome a wide range of problems associated with the requirement of miniaturization of medical devices and the trend towards less invasive and less traumatic procedures. Devices made of Ti-Ni alloys with shape memory effect and pseudo elasticity provides unique combination of physical and mechanical properties.

In the development of power structures of implants the most important property of Ti-Ni alloys is the superelasticity. It corresponds to the biological tissue hysteresis behavior. Their mechanical properties are characterized by a delayed reaction to external influences. The property of living tissues reacting to any mechanical impact with a delay develops by the fact that when tissues are under load, an increase in resistance is observed. But after the load is removed, the resistance decreases not immediately, but with a delay [1]. The similar loading behavior of Ti-Ni alloys makes them ideal implants, especially given the unique corrosion resistance of these alloys, including in organic environments[2].

The development of methods for surface modification of alloys is also promising [3-6], which also makes it possible to manage the concentration of Nickel in the near-surface layer while maintaining the characteristics of the thermomechanical behavior of the product [7-11].

Alloys with shape memory and pseudoelasticity are also used in the design of devices for percutaneous transvenous implantation. Due to the effect of superelasticity, the use of Ti-Ni alloy in such devices as a power element makes it possible to correct the pathological expansion of anatomical structures in the area of the mitral valve of the heart [12, 13], which

* Corresponding author: tatianamorozova@misis.ru

is relevant at the present time. During the endovascular implantation method, the power-unit device experience significant deformation when placed in the delivery catheter. But when removing the external load, the product must completely restore its preset shape due to implementation of the superelasticity effect. This happens when the implant is removed from the delivery catheter.

The operational properties modeling and development of an appropriate technology for pretreatment of the device material will make it possible to forecast the required level of operational characteristics [14-18], which must be provided for reliable operation of the power element. The purpose of this study was to explore the possibility of using TiNi alloys with shape memory and pseudoelasticity as an implantation material, that provides correction of abnormal enlargement of the anatomical structures of the mitral valve of the heart, as well as to establish the optimal mode of thermocyclic processing of the power-unit device material.

Since the shape of the device's power element was a rod bent in the form of a spiral, and the device must endure significant deformation during endovascular implantation into the coronary sinus, a deformation testing scheme was developed. The evaluation of the device's performance characteristics was based on the results of cyclic loading at tension and bending, which make it possible to assess the effect of different stress states during deformation on mechanical properties and the process of shape restoration.

2 Experimental Procedure

In the manufacture of device was used the wire on the base of nitinol Ti49.4Ni50.6 with shape memory effect and pseudoelasticity, which is used in medicine. The studied samples were obtained by warmly drawing hot-rolled TiNi rods up to a diameter of 0.9 mm.

The temperature of the phase transitions was determined by differential scanning calorimetry [19]. Upon cooling, the martensitic transformation of B2→R→B19' occurs in the alloy. During heating, a transition of B19'→ B2 should be expected, however, up to -110^{oC} it is not possible to completely finish the martensitic transformation of R→B19', therefore, during heating, the transition of R→B2 occurs simultaneously. As is known, the effect of deformation in such alloys stimulates the transition B2→ B19', suppressing the formation of the R-phase.

The temperatures corresponding to the beginning and end of the martensitic transformation of the alloy are shown in Table 1.

Table 1. Transformation Temperatures for $Ti_{49.4}Ni_{50.6}$ Alloy

Transformation	°C	Transformation	°C
Rs (start of B2→R)	+4 ± 2	A_f' (finish of R→B2)	+1 ± 2
Ms (start of R→B19')	-56 ± 2	A_f (finish of B19'→B2)	-9 ± 2
M_f (finish of R→B19')	<-110	As (start of B19'→B2	-33 ± 2

The deformation cycling was performed using Instron 3365. The length of samples was 70 mm, deformation rate was 0.0017 c^{-1}.

The method of measuring the deformation and stress of alloy samples under tensile conditions is shown in the deformation diagram, according to which the following characteristics were determined: the amount of residual deformation, the amount of total deformation, the width of the superelastic hysteresis loop (Fig.1).

When designing the power element of the device, it was considered that during endovascular implantation (bending, local stretching and compression), the temperature range of the martensitic transformation may shift, and residual deformation may appear after

removing the external load. It was estimated the amount of necessary deformation for the studied rods with a diameter of 0.9 mm, under conditions that they were placed in the delivering catheter 12 Fr and 15 Fr.

During endovascular implantation, the device may be subjected to deformation not exceeding 3%. The testing of samples to determine the mode of deformation cycling was carried out by stretching deformation by 7%.

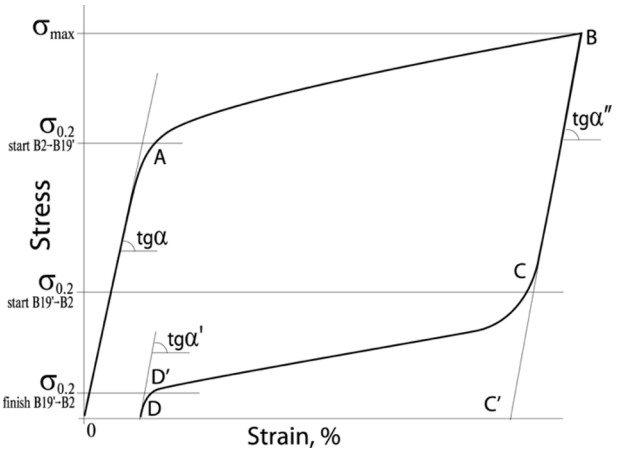

Fig.1. Schema of Mechanical Properties Measurement. DC' – deformation after stress induced transformation; OD - unrecoverable strain; tg α - The effective module of elasticity (start of loading); tg α' - The effective module of elasticity (finish of unloading); tg α" - The effective module of elasticity (start of unloading)

The effect of cyclic loading on the mechanical properties of a power-unit element having the effect of superelasticity was studied using deformation curves. The deformation curves of the studied alloy have a typical hysteresis appearance, typical for alloys with the effect of superelasticity. According to numerous literature insights, the upper branch corresponds to the direct martensitic transformation B2→ B19', the lower one to the reverse transformation. In this case, the direct transformation proceeds by the mechanism B2→R →B19', respectively, the reverse martensitic transformation takes place in the reverse sequence. After the first loading cycle, the samples were deformed by stretching by 7%.

Then, after the load is removed and the reverse martensitic transformation was made, the induced deformation is restored, but in this case not in full volume (Fig.2), Incomplete restoration is explained by the presence of a martensitic phase in the structure of the samples in the initial state.

The curve of the first cycle has a fairly wide hysteresis, which characterizes dissipative losses during deformation during forward and reverse martensitic transformations during the movement of the interphase boundary [1, 2].

Diagrams of cyclic loading of samples are shown in Fig.3. Cyclic tests were carried out under cyclic tension until 7% deformation value was reached for each cycle and the load was subsequently removed. As a result of the tests, during each cycle of stretching and relieving the load, residual deformation accumulated. The maximum value of the total deformation after all cycles was 3%. It should be noted that the increment of residual deformation per cycle increased significantly during the first 10 cycles, and after 20 cycles it practically did not change (Fig.4). Thus, such preliminary cycling will stabilize the reversible deformation of the force element, ensuring the restoration of shape after the necessary deformation in the delivering catheter.

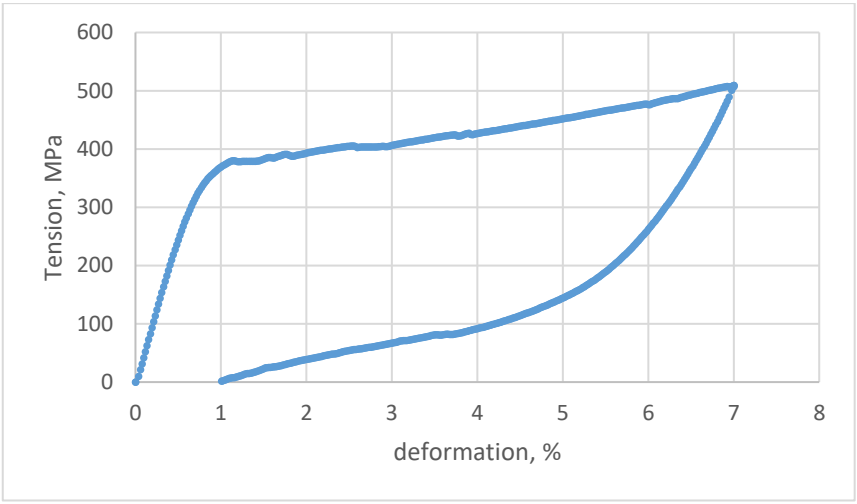

Fig.2. Deformation stress dependence of a TiNi alloy sample at a deformation rate of 0.0017 s^{-1}

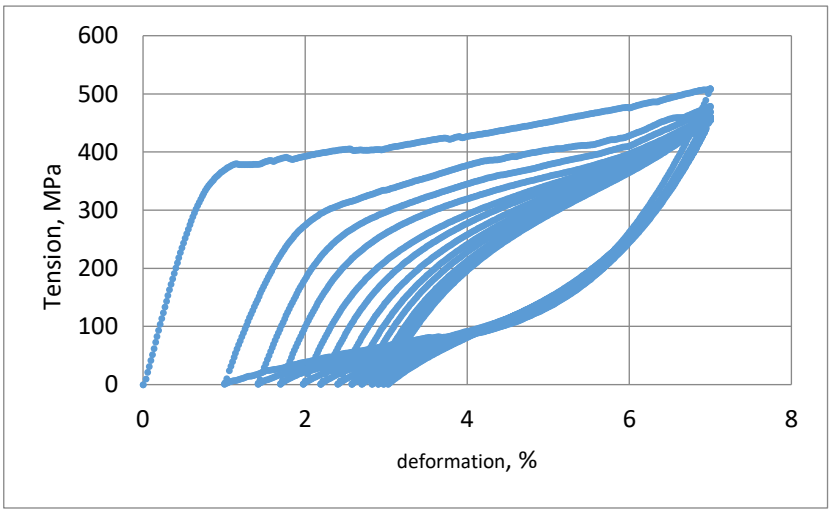

Fig.3. Deformation stress dependence after cyclic loading of Ti-Ni alloys at a deformation rate of 0.0017 s^{-1}

At the same time, during the deformation cycling, a change in the shape of the deformation curve was observed. With an increase in the number of cycles of direct and reverse martensitic transformations, a gradual decrease and narrowing of hysteresis was stated, which characterized a decrease in the dissipated energy and its tendency to a certain stable value (Fig.5.), which may indicate structural stabilization of the interphase boundaries, further ensuring the reversibility of deformation [20-22].

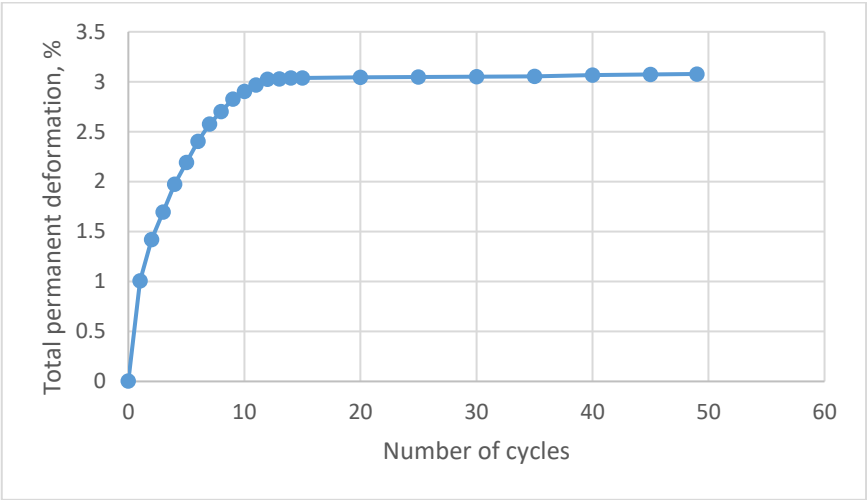

Fig.4. Change in the total residual deformation after cyclic loading of the Ti-Ni alloy

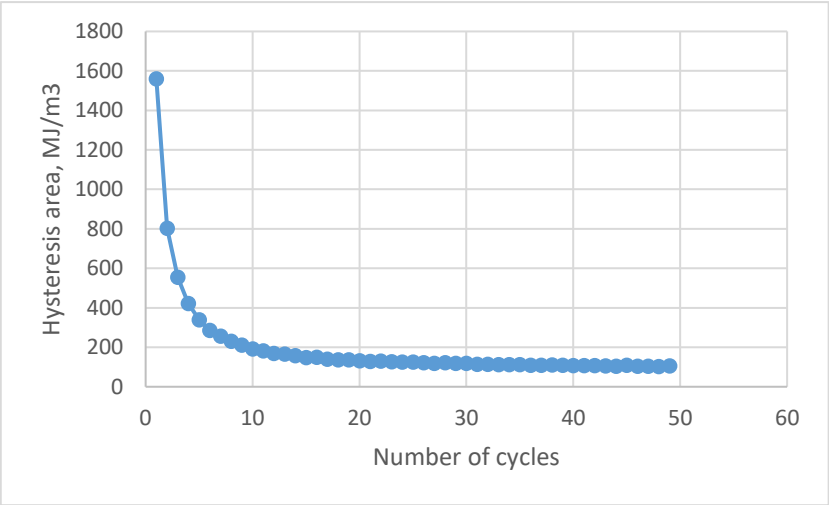

Fig.5. The change in the specific energy dissipated during the cycle of direct and reverse martensitic transformations, depending on the number of cycles of Ti-Ni alloy

3 Results

1. Preliminary cyclic deformation ensures the mechanical properties spectrum stabilization which guarantees a constant shape of the implant after its installation. The results were used in the development of a power-unit prototype for overcoming mitral regurgitation device.

2. The mode of deformation cycling is proposed.

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