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# On the Nitinol properties improvement after electrochemical treatments

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**ABSTRACT**

Shape Memory Alloys (SMAs) are a group of intermetallic compounds, which can undergo deformation at one temperature, and then recover their original undeformed shape upon heating above their transformation temperature. Superelasticity occurs just above the alloy's transformation temperature in a very narrow range. In this case no heating is necessary to cause the deformed shape to recover upon load relieve to its original undeformed shape. It has to be emphasized that nitinol exhibits enormous elasticity when compared with other medical metal alloys. In recent years, the use of nitinol (NiTi), almost equiatomic binary (50:50 ratio) intermetallic compound of nickel and titanium, has been steadily growing, particularly in medical and dental devices markets. However, broader and further application of nitinol has been slowed down by leaking nickel and unavoidable inclusions during producing in this compound. This work is to present some electrochemical treatment methods in view of reducing of both these phenomena. It appears that changing electrical conditions of electropolishing (EP) above the plateau region (EP+) may improve the quality of surface obtained on NiTi of over 60% in comparison with as-received (AR) nitinol part. What's more, introducing a magnetic field into the electrolysis system results in numerous positive features of nitinol surface and increase of mechanical properties. Thus the magnetoelectropolishing (MEP) process appears to increase higher the fatigue resistance of the treated NiTi part. The experiments carried out on

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chirurgical needles show an unusual triple (and higher) growth in resistance to bending until fracture. Further increase in fatigue resistance is usually limited by different size inclusions appearing on the nitinol part surface under magnetoelectropolishing (MEP).

***Keywords*****:** Nitinol, SMA, inclusions, electropolishing (EP), magnetoelectropolishing (MEP), fatigue resistance

## 1. INTRODUCTION

Nitinol as a shape memory alloy (SMA) is remarkable material opened upon a wide range of uses. In fact, Nitinol is an intermetallic compound formed of nickel and titanium. Some of its major applications are in medicine to produce medical devices. Its unique properties allow minimally invasive surgery using stents and implants to improve quality of life for patients.

Nitinol was an unexpected discovery by William Buehler from the US Government Naval Ordnance Laboratory in the 1960s. He named the material 'NiTiNOL' which stood for Nickel Titanium Naval Ordnance Laboratory [1]. They were developing fatigue, wear and impact resistant materials for military purposes. During studies they discovered the reason behind the behaviour of the material which was due to atomic rearrangement or phase changes at different temperatures while the material was still a solid.

Melting methods, type of raw materials and processing techniques result in impurities which lead to the formation of non-metallic inclusions which are critical to control [2-4]. The oxygen, nitrogen and carbon content in the melt form titanium oxides, nitrides and carbides. They are hard inclusions acting as discontinuities in the matrix. These have been the subject of numerous studies on device failure and fatigue strength [5-9].

In recent years, the use of nitinol (NiTi) almost equiatomic binary (50:50 ratio) intermetallic compound of nickel and titanium has been steadily growing, particularly in medical and dental devices markets [9-13]. However, broader and further application of nitinol [14-20] has been slowed down by two factors:

- a very high nickel content [21-23]
- misunderstanding of the role of inclusions, which presently are unavoidable in this intermetallic compound [8, 24-29].

Biomaterial surface alteration, which must include the influence of sterilization on the biomaterial, is the very important way to tailor post implantation interaction between biomaterials and biomolecules adsorption and further cellular processes.

The main focus of this work is the development of new electrochemical processes, which will be able to improve fatigue resistance, to cost effectively and reliably test nitinol for surface inclusions, alter surface in the direction of improved bio- and haemocompatibility, and simultaneously sterilize nitinol biomaterial.

A representation of the shape memory and superelastic effect [1, 14, 23, 30] with specific reference to Nitinol is seen in Figure 1. Since the material is equiatomic, one sphere in the crystal structure represents nickel and the other represents titanium.

The structure at the top (Fig. 1a) represents the crystal structure of the material in the stable or austenitic state. This is when the material is above the austenite finish $A_f$ (Fig. 2).

When the material is cooled below the martensite start temperature, $M_s$, it starts transformation into twinned martensite (like a herringbone) shown in the lower right structure, (Fig. 1b). Below the martensitic finish ($M_f$) temperature, the material is completely martensitic. This martensitic transformation is called thermally induced or twinned martensite. When twinned martensite is subject to stress, it transforms into deformed or detwinned martensite, (Fig. 1c).

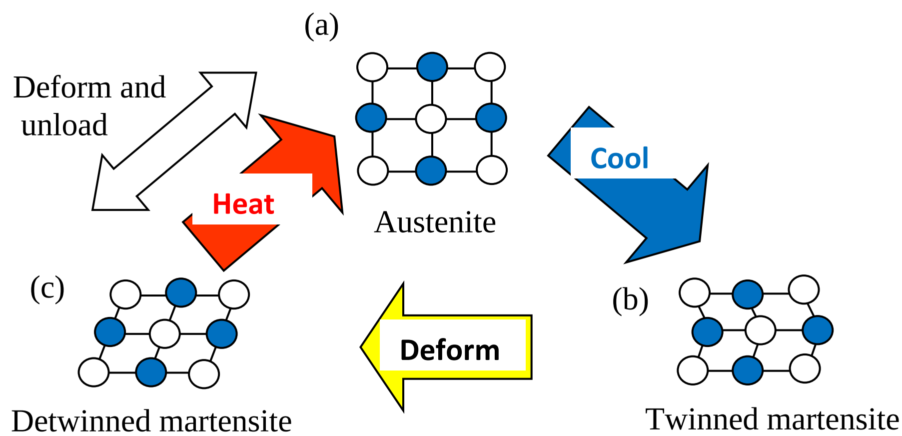


**Fig. 1.** Schematic illustration of shape memory effect

The shape memory effects can be divided into one-way and two-way shape memory effects. The two-way effect appears very rarely. In the one-way effect nitinol sample is in full austenite phase in the temperature above $A_f$ (austenite finish). When the sample is cooled below the temperature $M_s$ (martensite start) the phase transformation takes place and austenite is replaced with twinned martensite. The phase change continues until $M_f$ (martensite finish) temperature is reached and at this stage the intermetallic compound is composed exclusively of martensite (monoclinic crystal structure). Mechanical loading of the martensite causes the appearance of detwinned martensite, which shows a residual strain after removal of the load. After SMA has been distorted, the original shape can be recovered by heating the detwinned martensitic sample from the austenite transformation start temperature $A_s$ (austenite start) to the $A_f$ temperature, where the alloy again reaches the fully austenite state [31]. $A_f$ is the upper end of the transformation temperature range. The heat transferred to the material is the power driving the molecular rearrangements of the intermetallic compound. Those atomic rearrangements are of displacive and not diffusional nature. This means that the atoms are rearranged in another crystal structure during phase transformation, but the chemical composition of the matrix remains the same.

Two-way shape memory is also a martensitic transformation phenomenon. In this type of transformation the sample has two different customized shapes, martensite and austenite. In contrast to one-way shape memory, in two-way memory shape the change of temperature produces a change in sample shape without any mechanical loading [31].

Pseudoelasticity in nitinol occurs when nitinol is completely in austenitic phase (temperature higher than $A_f$). Unlike the shape memory effect, pseudoelasticity occurs without a change in temperature but only under the applied load. When the load on nitinol in austenitic phase is increased, austenite transforms into martensite (Fig. 2). The loading is absorbed by softer martensite [32]. But when the load starts to decrease, martensite immediately transforms to austenite because temperature is still above $A_{f.}$. The material springs back to the original shape.

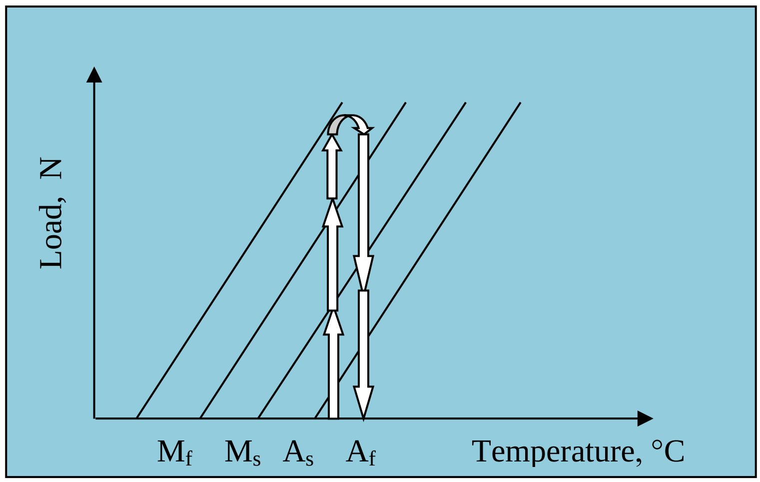


**Fig. 2.** Load diagram of pseudoelasticity of nitinol

## 2. NITINOL FOR MEDICAL APPLICATIONS

In recent years, the use of nitinol has been steadily growing, particularly in medical and dental device markets. Nitinol's popularity is due to its unique and unusual properties of thermal shape memory, pseudoplasticity (strain recovery), good dumping properties and very good biocompatibility when surface is adequately finished. The very good ductility and malleability allow the material to be produced in many forms such as: wires, tubes, sheets, roods or bars. Excellent ductility allows nitinol to be drawn as wires and tubes with such small diameters that they can be used in production of microscopic vascular devices utilized in treating vasculature of brain.

Generally, nitinol-based medical devices can be divided into two groups: implantable and surgical tools. Implantable devices consist of vascular (peripheral stents, aneurysm coils) and cardiovascular (cardiovascular luminal shields, heart valves, septal occluders). The surgical tools and materials include: stone and blood clots retrievers, endoscopes, cutting blades, guide-wires, arthroscopic tools, etc. One particular group of nitinol medical devices which can be classified in both ways is nitinol inferior vena cava filters (IVC-filters). If the filter is retrieved from the vein after its role of catching thrombus is finished, it can be classified as surgical tool, but if it is left permanently it should be classified as an implantable medical device. It is worth to note that IVC nitinol filters permanently left in veins are responsible for many undesirable events such as tilting, migration and fracture [33, 34].

Nitinol's unique mechanical properties of pseudoelasticity (stress-strain behavior) when loaded and unloaded are very similar to that of human bone (Fig. 3). Its low elastic module between 20 and 90 GPa is close to that of cortical bone between 15 and 25 GPa [6, 7, 35]. Nitinol is the only orthopaedic implantable metallic material which yields in response to pressure. This feature makes it very suitable for use in bone-fixing devices such as bone plates, staples, spine fracture fixation devices, maxillofacial and dental implants and intramedullary nails.

Nitinol also found its permanent place in dentistry as the material of endodontic rotary files and arch-wires. Endodontic rotary files endure severe cyclic fatigue loading under very high rotation speed and sometimes almost 90° angle for several hundred revolutions.

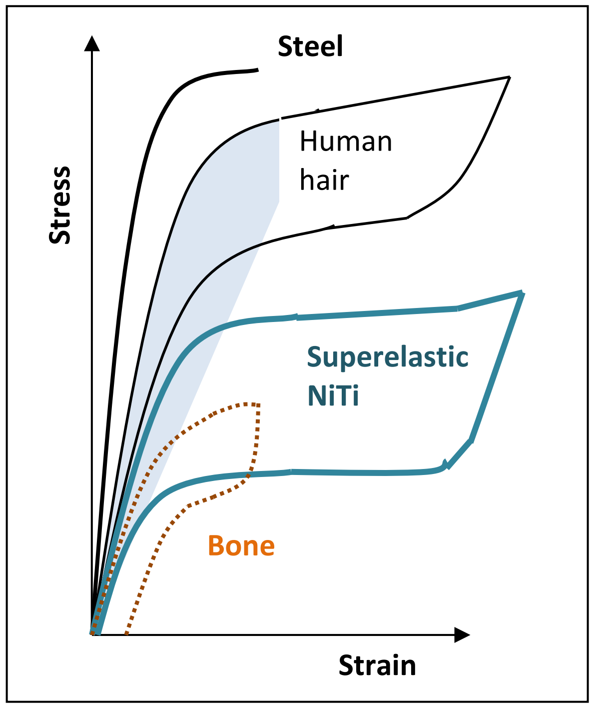


**Fig. 3.** The stress-and-strain hysteresis loop of superelastic nitinol in comparison to steel and some biological materials. When stress is released the shape of material is recovered

It is very important to find the best possible way to finish nitinol implantable devices after all production steps (machining, drawing, shape seating and aging and oxide removal, including sterilization), and establish one binding protocol. The tendency to fracture is the heel of Achilles' of nitinol vascular implantable devices.

## 3. ELECTROCHEMICAL TREATMENTS

In the absence of any mechanical surface treatment protocol the treatment methods used are of proprietary origin. They include sanding, grinding, high luster-polishing followed by wiping or ultrasonically bathing of residues with water, detergents, organic solvents, etc. Surfaces treated by these methods lack reproducibility in corrosion resistance [36], the problem which is usually solved by electrochemical treatments, such as electropolishing (EP) [15, 23, 26, 29, 30, 41-43] and magnetoelectropolishing (MEP) [15, 19, 20, 26, 29, 37-40].

Currently, electropolishing process, followed by sterilization, is the gold standard of finishing nitinol implantable devices such as: stents, heart frame valves, IVC filters, etc.

Electropolishing is a hundred years' old electrochemical process which uses electrolysis principles. The metal object (in this case nitinol) which has to undergo electropolishing is connected to the positive terminal of DC (direct current) power supply submerged in appropriate electrolyte in electrochemical cell with negatively connected cathode. When current is applied to the cell the anode (metal object) starts to dissolve under controlled conditions. Upon process termination the surface of treated metal object becomes smoother, more corrosion resistant and in case of implantable devices more biocompatible.

## 4. FATIGUE RESISTANCE

Corrosion resistance, biocompatibility and fatigue resistance are some of the most important features of high importance in biomaterials to be safely used for human applications. First two properties have been studied for years with quite good results [2, 5-7, 9-13, 15-17, 20, 28-31, 37-41]. Mechanical properties were also studied, with the results dependent usually on the surface finish of parts and implants, achieving 25 to about 50% improvement measured against as-received (AR) nitinol parts. Cyclic fatigue testing of nitinol needles used during arthroscopic meniscus repair surgery was carried out using the Mark 10 ESM 301 special tester (Fig. 4), with a custom-made needle guide.

**Fig. 4.** Mark 10 ESM 301 tester

Fatigue resistance of nitinol parts, surgical tools and implants, can be highly improved by the process of magnetoelectropolishing (MEP). As the example, the needles 50 mm long, of flat cross-section of 1.42×0.29 mm were investigated in a specially prepared slideway using 10 ESM 301 tester (Fig. 4). The introductory results show over triple up to over 7 times higher number of cycles before fracture/distructure. The average number of test cycles until fracture for the magnetoelectropolished needles is almost five times greater than for the electropolished one (Fig. 5). Nonetheless the MEP finish poses the highest standard deviation among other finishes. The maximum bending cycles reached for this finish (220) is more than double the minimum one (97). So the big spread is almost five times larger than that of the spread for the two other electropolishing finishes (EP and EP+). However, the minimum number of cycles until fracture in the MEP group is triple of that of AR samples and almost double than the ones in both EP groups. This can only be explained by differences in properties of titanium oxide created by MEP and EP processes. MEP creates more stoichiometric, more homogeneous titanium oxide, which poses better elasticity and by that improves fatigue resistance during the bending test. The big spread can be explained by the intermetallic inclusions on the surface.

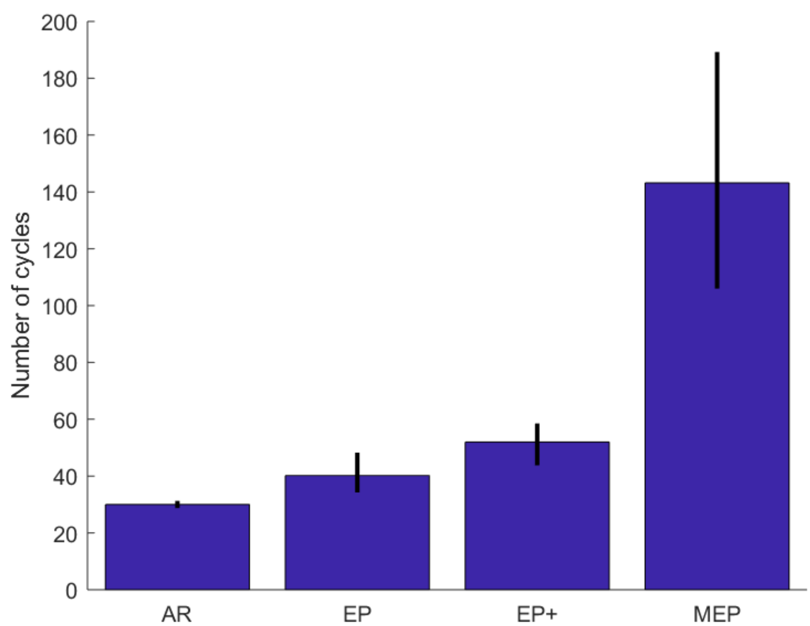


**Fig. 5.** Chirurgical needle bending cycles till fracture after electropolishing processes

## 5. DISCUSSION

During the test, the needle bends 90° in one direction and 70° in opposite. The second part of the needle consists of a 205 mm rod of 1.53 mm diameter, which pushes and pulls the needle during surgery. The tested needles were pushed and pulled from a custom-made guide with 178 mm/min speed till fracture. It also has to be pointed out that the needles electropolished in the transpassive region (EP+) withstand more numbers of test cycles before fracture than needles electropolished in the standard way (EP), namely in the plateau region, below oxygen evolution regime.

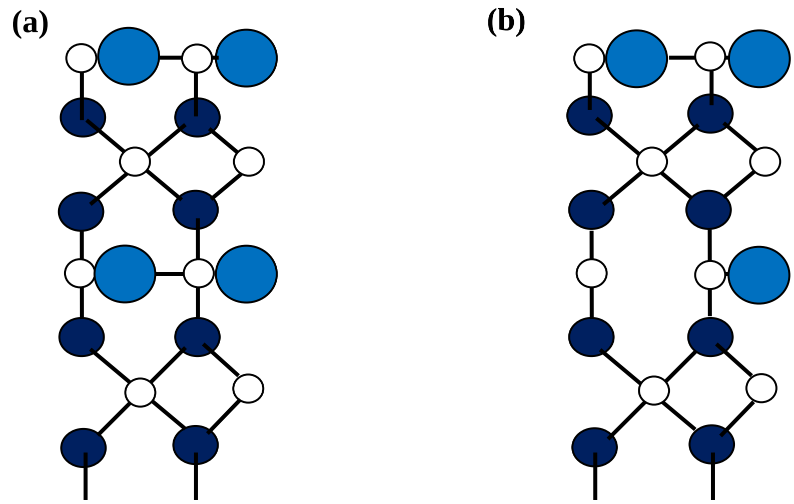


**Fig. 6.** Stick-and-ball diagram of perfect crystal lattice of $TiO_2$ **(a)**, and defected crystal lattice with missing oxygen ion in the second row **(b)**

One of the most probable reason for highly improved – for several times – properties of MEP nitinol samples are changes occurring between crystal lattices of $TiO_2$ and defected crystal lattice with missing oxygen ion (Fig. 6b). Such a change represented by stick-and-ball diagram (Fig. 6) results in a considerable difference between the mechanical properties in the number of cycles, as presented in Fig. 5.

## 6. CONCLUSION

In metallurgical sense nitinol is not an alloy but intermetallic compound, which means that it is composed of definite proportion of elemental metals: titanium and nickel with very strong internal order and mixed metallic and covalent/ionic bonds. This strong metallic bonding of nickel and titanium elements makes nickel leaching from nitinol implanted device more difficult than from other metallic materials such as, for example, stainless steel, which from metallurgical point is an alloy. However, this statement is valid only for perfectly homogeneous nitinol.

The experiments presented, and longstanding professional experience provide adequate justification to confirm that the fatigue resistance of nitinol parts, surgical tools and implants, can be highly improved by the process of magnetoelectropolishing (MEP). Fig. 4 clearly shows that electropolishing processes in the magnetic field can considerably improve the bending cycles till fracture. Further research is needed to clarify in more detail the effective number of cycles, if it is only 3 times, or over 7 times higher than that after a standard surface treatment.

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