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Characterization of titanium–niobium orthodontic archwires used in orthodontic treatment

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The titanium-niobium (TiNb) alloys are known as superconducting materials and they are used for the production of superconducting wires. They are also widely applied in the medicine, for the needs of orthopaedic reconstructive surgery, implantology etc. due to their properties, as high biocompatibility and high corrosion resistance. Recently, this material has been incorporated in orthodontic braces as finishing archwire during the process of orthodontic treatment. On the market exist different types of archwires, developed by many producers, which obey specifications necessary to apply them during orthodontic patient treatment. But properties of these archwires are deteriorating with time. To apply them properly it is needed to find the reasons of such deterioration and the methods to register it. We try to gather such knowledge – this time for some archwires produced by the ORMCO Company – TiNb ones.

The analyses were carried out by the independent techniques: X-ray diffraction analysis (XRD), Scanning Electronic Microscopy (SEM), Energy Dispersive Spectroscopy (EDX) and Differential Scanning Calorimetry (DSC) and Physical Property Measurement System (PPMS). The composition of the wires was established to be: 57,15wt% Ti and 42,85wt% Nb. The low temperature magnetic studies revealed that the material possessed superconducting properties with transition to zero resistant state at about 10K and above this superconducting transition temperature, the material was paramagnetic. From DSC results it has been concluded that there was no thermal transition of the alloy in the temperature range from -50ºC to +50ºC. The received knowledge will allow to estimation of the safe TiNb archwires application in orthodontic treatment.

Keywords: TiNb, orthodontic archwires, XRD, SEM, DSC

INTRODUCTION

Titanium – Niobium alloys are well known as cryogenic materials [1], exhibiting superconductivity they are used in superconductor production [2-3]. Due to this property they find an application in magnets for nuclear magnetic resonance imaging systems [4]. This alloy is composed of two of the five elements (Nb, Ta, Ti, Zr and Pt) which do not cause tissue reactions [5-6], it has excellent corrosion resistance and biocompatibility [7]. This is the reason for its wide use in medicine as a substituting material in reconstructive orthopedic surgery and implants. Due to their low elasticity modulus titanium alloys are beneficial for distributing the stresses between bones and implants [8]. With addition of Nb elastic modulus is further reduced and corrosion resistance is improved [9].

Orthodontic archwires, as a material placed in the oral cavity, in addition to good mechanical properties must also be biocompatible. A number of cases of allergic reactions to orthodontic materials are known and are caused primarily by the release of Ni ions [10-11]. In these cases use of an alloy which does not contain nickel is recommended [12-13]. During the aligment phase of treatment composite arches [14] may be used or ion-impacted Ni-Ti arches in which an amorphous surface layer is formed, inhibiting corrosion and release of Ni ions [15]. After the aligment phase of treatment

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TMA arches containing Ti and Mo [16] are most commonly used. As an alternative to this non nickel-containing alloy an innovative Ti-Nb alloy arch is proposed [17] for this there is little available data. This archwire possesses extremely high corrosion resistance thanks to a surface layer of Nb_2O_5, inhibiting the action of fluoride ions responsible for quick corrosion in the oral cavity [18]. In light of its mechanical properties [19] and high friction coefficient [20] it is suitable for the finishing stages of orthodontic treatment, in which bracket sliding is not needed and applying finishing bends is necessary.

The aim of this study is to make characterization of the microstructure, chemical composition and investigation of magnetic properties of two types, as-received TiNb wires, produced by the ORMCO Company. The received knowledge will allow to estimation of the safe TiNb archwires application in orthodontic treatment.

MATERIALS AND METHODS

TiNb orthodontic archwires produced by ORMCO Company, CA, USA, with two different cross sections – 0.019x0.025 inches^2 (0.48 x 0.63 mm^2) and 0.017x0.025 inches^2 (0.43 x 0.63 mm^2) were investigated. Cut wire pieces of both type as-received TiNb archwires were studied with different techniques, as X-ray Powder Diffraction (XRD), Scanning Electron Microscope (SEM), Energy-Dispersive X-ray microanalysis (EDX), Differential Scanning Calorimeter (DSC) and Physical Property Measurement System (PPMS).

To determinate the structure of the material it is used X-ray Powder Diffractometer X'Pert Pro, with low-temperature nitrogen blower attachment Oxford Cryosystem and high-temperature closed attachment HTK 1200 from Anton Paar. The SEM tests were performed using Philips 515 Scanning Electron Microscope with accelerating voltage 0.2 to 30 kV. The composition of the archwires was obtained by EDX method using Bruker Esprit 1.8 system. Minimum acceptable accelerating voltage of 20 kV was applied. Quantification of the EDX results was performed by interactive PB-ZAF standardless method. The DSC analyses were performed using a differential scanning calorimeter Perkin - Elmer – 8000 with TGA attachment model PE-TGA4000. Before introducing the sample in the DSC apparatus for each individual test a calibration with indium was made. The temperature range of DSC is from - 170^{o}C to +600^{o}C. The samples were scanned from -50^{o}C to +50^{o}C and back to -50^{o}C with 20^{o}C per minute. The magnetic properties of the samples were tested with PPMS from Quantum Design, Inc. which conducts research in the temperature range 1.9K-400K in a magnetic field up to 9T.

RESULTS AND DISCUSSIONS

Fig. 1 displays the diffraction pattern of TiNb (0.019x0.025 inches^2) archwire. The registered diffractions picks demonstrate presence of Ti and Nb, corresponding with the results reported by Mi-Kyung Ha et all. [21], whose indicate that TiNb alloys are more sensitive to the Nb content.

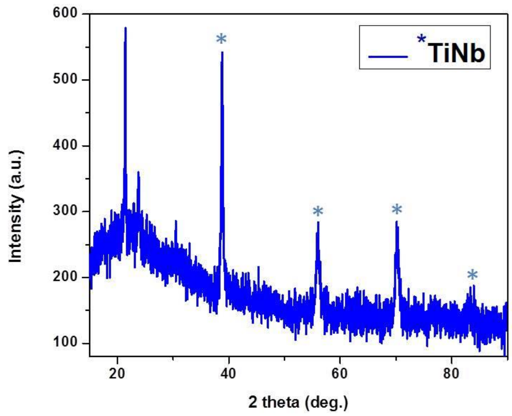
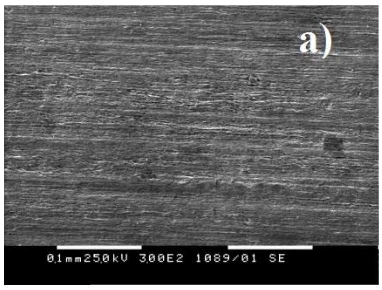


Fig. 1. XRD pattern on TiNb archwire 0.019x0.025inches^2



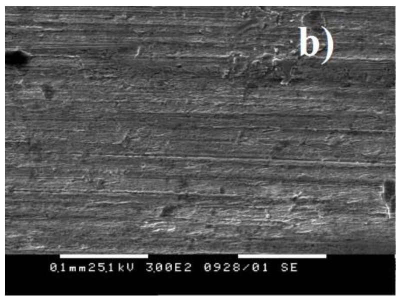


Fig. 2. SEM images of as-received archwire 0.017x0.025 inches^2 (a) and 0.019x0.025 inches^2 (b)

Report about the change in the surface morphology of the archwires with both dimensions is obtained by SEM. The SEM micrographs of two types as-produced TiNb archwire are presented in

Fig. 2a for 0.017x0.025 inches^2 and Fig. 2b for 0.019x0.025 inches^2. Typical surface morphology of wires produced of metal alloys by extrusion is clearly seen. Some defects on the surface, predominantly in the case of 0.019 x 0.025 inches^2 archwire are detected. They might be very deteriorating for applied archwires, as existing micro-cracking and voids might deepen during patient curing and to worse mechanical properties of the archwires (elasticity and shape memory).

For further information of the composition of the surface topography is used EDX. This method allows quantitative identification of the chemical composition by elements. For higher certainty EDX was done in 4 parts of each investigated piece of the both as-received TiNb archwires (0.019 x 0.025 inches^2; 0.017 x 0.025 inches^2). The analysis confirms that the main components of the archwires are only Ti and Nb (Table 1. and Table 2.).

Table 1. Elements content in as-received TiNb archwire (0.019 x 0.025 inches^2).

Spectrum	1	2	3	4
Elements	Wt%	Wt%	Wt%	Wt%
Titanium	57,15	57,52	57,05	57,11
Niobium	42,85	42,48	42,95	42,89
Total	100,00	100,00	100,00	100,00

Table 2. Elements content in as-received TiNb archwire (0.017 x 0.025 inches^2)

Spectrum	1	2	3	4
Elements	Wt%	Wt%	Wt%	Wt%
Titanium	57,26	57,21	57,87	57,57
Niobium	42,74	42,79	42,13	42,43
Total	100,00	100,00	100,00	100,00

There is no presence of additional elements as chromium, cobalt, copper, which are registered on the other popular archwires as NiTi and CuNiTi [22 - 25]. In the orthodontic archwire with the (0.019 x 0.025 inches^2) dimension, has average value of the elemental composition with approximately Ti 57.20 wt. % and Nb 42.79 wt. % . Respectlivity, for the (0.017 x 0.025 inches^2) archwire, Ti 57.47 wt. % nd Nb 42.52 wt. % .

In orthodontics different archwires are used, in some of them (for example heat-activated) the temperature changes lead to changes in the mechanical properties of the archwires. These archwires at body temperature have an austenitic structure and possess high elasticity and shape memory. With cooling of the archwires out of the patient's mouth with cryogenic spray to -50 ^{o}C, the structure of the material changes to martensitic and the material becomes flexible, easily deformable and adaptable. After placing it in the mouth at 37 ^{o}C, the archwire gradually warms up and changes the structure into austenitic again. That is why it is useful to investigate the thermal behavior of TiNb archwires, which are a new material applied in orthodontics. In this study DSC method is used to verify if there is a transition temperature for the austenitic, martensitic, and rhombohedral (R) structure phases in representative as-received TiNb archwires. For DSC analyses very small samples were prepared and a special test cell is able to cool and heat slowly the sample with a very precise rate. During the cooling or heating process, the cell indicates whether the sample is either giving off more heat or absorbing more heat in exothermic or endothermic reactions, respectively [26]. On Figure 5 are shown DSC curves, which demonstrate that there is no thermal transition on the both 0.019 x 0.025 inches^2 (Fig. 3a) and 0.017 x 0.025 inches^2 (Fig. 3b) archwires, measured in the temperature range from -50 ^{o}C to +50 ^{o}C.

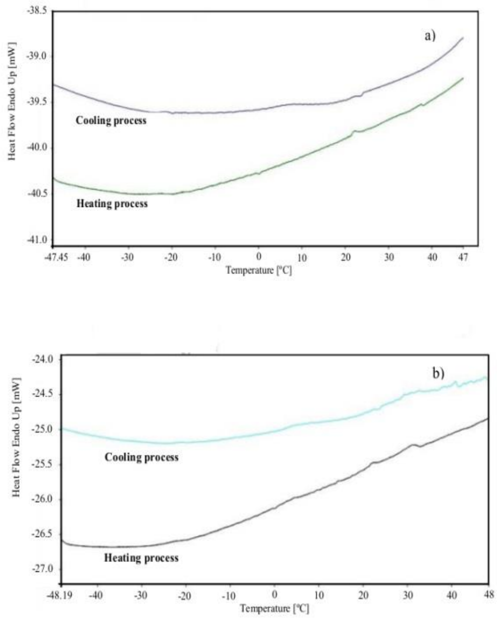


Fig. 3. DSC analysis on as-received TiNb archwires 0.017x0.025 inches^2 (a) and 0.019x 0.025 inches^2 (b)

It is known that the metals Ti and Nb become superconducting at sufficiently low temperatures. Solid solution of Ti and Nb, which can be obtained for the whole range of compositions, is also superconducting and its properties depends mainly on composition, structure and heat-treatment, respectively. Allowing α–titanium precipitate in the volume of TiNb solution leads to obtaining of superconducting material with very high current density values. This material is used for wire production necessary for superconducting magnets [27]. In order to obtain more information about archwire, made from TiNb alloy, used in orthodontic medicine we studied also their magnetic properties at low temperatures. The results shown on Fig. 4 demonstrate that at about 10K real part of susceptibility changes abruptly from small negative value to strong diamagnetic one, proving that material is superconducting.

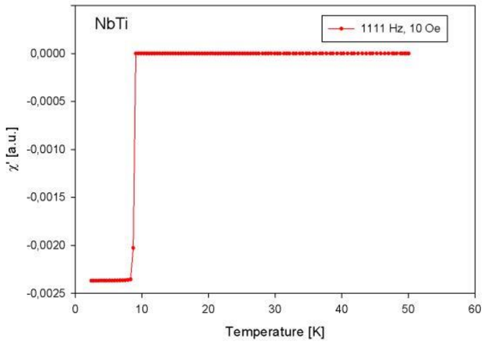


Fig. 4. Temperature dependence of the magnetic susceptibility of TiNb archwire (0.019 x 0.025 inches^2)

Although the material is superconducting it can carry very limited current, what can be seen from Fig. 5, where magnetic hysteresis measured at 2K is presented.

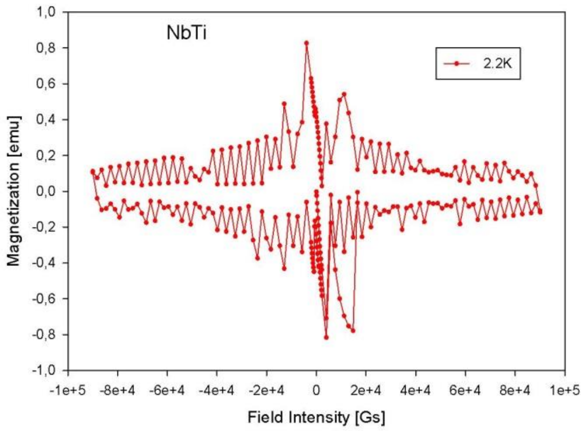


Fig. 5. Magnetic measurements on TiNb archwire (0.019 x 0.025 inches^2).

Above superconducting transition 10 K, the material is weakly paramagnetic. Future measurements will be made near room temperature where very precise DC magnetization measurements are needed.

These results for as-received TiNb archwires from ORMCO Company, will help the orthodonts to apply them successfully for patients treatment.

CONCLUSIONS

The manufacturers are experimenting with the elements of the alloys in order to establish and to improve the properties of the titanium archwires. That's why it is important to obtain the first profile of the elements and structure of TiNb archwires used for orthodontic appliances. Our results showed that the 0.019 x 0.025 inches^2 archwire has elemental composition, approximately Ti 57.20 wt % and Nb 42.79 wt%. Respectlivity, for the (0.017 x 0.025 inches^2) archwire, Ti 57.47 wt% and Nb 42.52 wt%. The DSC results made in the temperature range from -50^{0}C to +50^{0}C, showed that this archwire has no thermal transition. The magnetic properties of the archwires showed that this alloy is superconductor at 10K, and above superconducting transition the material is paramagnetic. The received knowledge will allow to estimation of the safe TiNb archwires application, as well as during the treatment.

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ОХАРАКТЕРИЗИРАНЕ НА ТИТАН- НИОБИЕВИ ОРТОДОНТСКИ ДЪГИ ИЗПОЛЗВАНИ ПРИ ОРТОДОНТСКО ЛЕЧЕНИЕ

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(Резюме)

Титан-ниобиевите (TiNb) сплави са известни като свръхпроводими материали и се използват за производството на свръхпроводими кабели. Поради техните свойства като висока биосъвместимост и висока устойчивост на корозия, те намират широко приложение в медицината, за нуждите на ортопедично-реконструктивната хирургия, имплантологията и др. Напоследък, този материал се използва за изработването на ортодонтска дъга, използвана в последния етап на ортодонтското лечение.

На пазара се предлагат различни видове ортодонтски дъги, от различни производители, които се подчиняват на спецификации, необходими, за тяхното прилагане по време на ортодонтското лечение. Но свойствата на тези дъги могат да се променят по време на лечението и за правилното им прилагане е необходимо да се намерят методи, чрез които тези свойства да бъдат проследявани. Целта на това изследване е да се получат данни за структурата, химичния състав и физичните свойства на TiNb ортодонтска дъга, произведена от фирмата ORMCO.