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COMPARISON OF NiTi ORTHODONTIC ARCHWIRES AND A DETERMINATION OF THE CHARACTERISTIC PROPERTIES

PRIMERJAVA ORTODONTSKIH LOKASTIH ŽIC NiTi IN DOLOČITEV ZNAČILNIH LASTNOSTI

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The aim of this paper was to analyse the characteristic properties of six different, commercially available nickel-titanium orthodontic wires with a diameter of 0.305 mm (0.014"). The characteristic properties were determined by using semi-quantitative EDX analyses, DSC analyses for a determination of the phase temperatures, and a tensile test to obtain the mechanical properties of the wires. The investigation of the chemical composition showed an equiatomic NiTi alloy. Analyses of phase temperatures showed that the nickel-titanium orthodontic wires were, in an austenitic microstructure, exhibiting a superelastic effect in the oral environment. The uniaxial tensile stress–strain curves showed different values for the beginning and the end of the transformation range during the loading.

Keywords: NiTi wire, shape-memory alloy, phase transformation, thermal analysis, tensile test

Prispevek obravnava analizo karakterističnih lastnosti 6 komercialno dostopnih nikelj titanovih ortodontskih žic, ki imajo premer 0,305 mm (0,014") v fazi pred njihovo uporabo v ustih. V teh okvirih so bile analizirane mehanske lastnosti z enoosnim nateznim preizkusom, za določitev faznih temperatur pa je bila izvedena DSC-analiza ter semikvantitativna EDX-analiza. Dobljene enoosne krivulje napetost – raztezek so pokazale različne vrednosti začetka in konca transformacijskega območja pri obremenjevanju NiTi-žic. Analiza faznih temperatur je potrdila, da imajo ortodontske žice avstenitno mikrostrukturo, s čimer kažejo superelastični efekt, potem ko so v uporabi v ustnem okolju. Krivulja napetost-raztezek pri enoosnem nategu je pokazala različne vrednosti za začetek in konec transformacijskega območja med obremenjevanjem.

Ključne besede: NiTi-žica, zlitina s spominom oblike, fazna transformacija, termična analiza, natezni preizkus

1 INTRODUCTION

The use of the NiTi shape-memory alloy (SMA) in orthodontics began in 1970 when Andreasen started advocating their use in this field. The first orthodontic wire on the market was made by the Unitek Corporation (now 3M Unitek, Monrovia, CA, USA).[1,2] Today, NiTi wires are extensively used in orthodontics.

SMAs are unique materials with an ability to recover their shape when the temperature is increased. These materials show two special behaviours: the *Shape Memory Effect* (SME) and the *Superelastic Effect* (SE) (**Figure 1**). Both effects are characterised by a martensitic phase transformation, which is caused by changing the temperature (shape memory) or the application of stress (superelastic effect) leading to changes in the phase or microstructure. The austenite phase is stable at high temperatures and small stresses, and the martensitic phase is stable at low temperatures and high stresses. These two phases have different crystal structures. The austenitic phase has a body-centered cubic crystal structure, while the martensitic phase has a monoclinic crystal structure. The transformation from one structure to the other does not occur by diffusion of atoms. The transformation is caused by a shear lattice distortion. The martensitic transformation is known as a diffusion-less transformation. The SME allows the alloy to return to its previous shape when it is heated above the A_f temperature. The SMA regains its original shape by transforming into the austenite phase. **Figure 1** shows the stress-strain-temperature diagram of a typical NiTi specimen tested under uniaxial loading in fully martensitic and

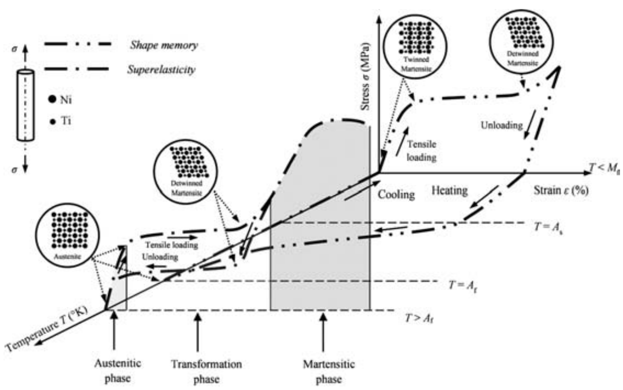

Figure 1: Stress-strain-temperature diagram exhibiting the Shape Memory and Superelastic effects of a NiTi alloy. Crystal transformation due to changing in temperature and stress.

Slika 1: Diagram napetost – raztezek – temperatura za zlitino NiTi z oblikovnim spominom in superelastičnim vedenjem. Kristalna pretvorba zaradi spremembe temperature in napetosti.

fully austenitic phases. The superelastic effect (SE) of the SMA is expressed above A_f. The SE is associated with a stress-induced transformation. **Figure 2** illustrates the superelastic effect. The SMA changes its microstructure from austenite to detwinned martensite when stress is applied. The SMA returns to its original shape when unloaded. The induced stress on unloading is less than that on loading, and the different paths between loading and unloading is referred to as hysteresis.[3–5]

The excellent biocompatibility and functional properties of SMA NiTi wires offer the possibility of their widespread use in orthodontics. In general, NiTi SMA wires show good corrosion properties. However, even though protective layers of TiO_2 are created on the surface of the wires, the release of nickel ions takes place. Biocompatibility studies suggest that SMA NiTi alloys have a low cytotoxity and genotoxicity.[6,7]

The orthodontic wire is a part of the fixed orthodontic appliance and its function is to deliver the optimal force on the tooth or the group of teeth. The optimum force used in an orthodontic treatment should be enough to produce tooth movement without tissue damage and pain. The NiTi SMA is used in the initial stage of the orthodontic treatment for teeth levelling and aligning. Low, continuous orthodontic forces are desirable, which is achieved by using wires with a low modulus of elasticity.[8–11] In this way, minimal periodontal tissue damage and maximum comfort for the patient are ensured.[12]

The objective of this paper was to analyse the functional and mechanical properties of different NiTi orthodontic wires used in orthodontic treatments. The correlation between the chemical composition and the functional properties, such as the temperatures of the phase transition and the stress-strain behaviour of wires, were investigated.

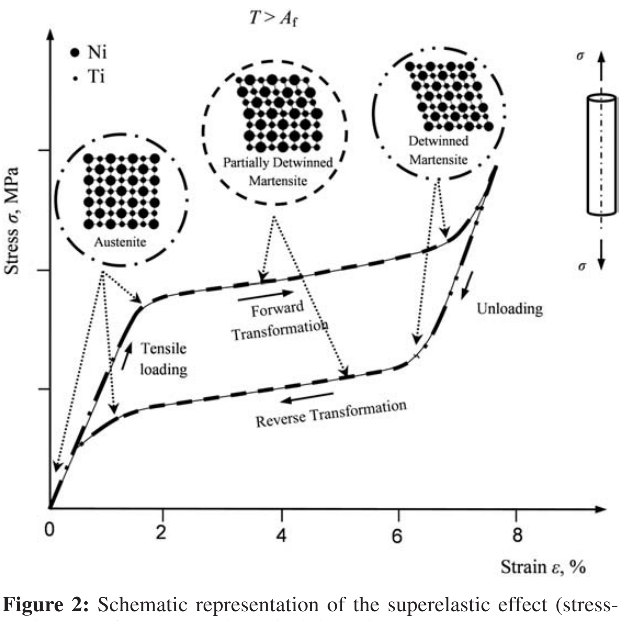

Figure 2: Schematic representation of the superelastic effect (stress-strain curve for SMA under uniaxial loading)
Slika 2: Shematski prikaz učinka superelastičnosti (krivulja napetost – raztezek za zlitine s spominom oblike pod enoosno obremenitvijo)

2 MATERIAL AND INVESTIGATION METHODS

New NiTi wires with a diameter of 0.305 mm (0.014"), taken from the original packaging, were used for the analyses (**Table 1**).

Table 1: Orthodontic wires from different producers included in the analyses
Tabela 1: Analizirane ortodontske žice različnih proizvajalcev

Orthodontic wire	Type of the wire
1	CuNiTi
2	Thermo-Active (NiTi)
3	NiTi
4	NiTi
5	Thermo (NiTi)
6	NiTi

2.1 EDX semi-quantitative chemical analysis

The SMA NiTi is characterized by an equiatomic proportion of nickel and titanium.[13] The chemical compositions of all six NiTi archwires were analysed on a longitudinal cross-section and on the surface of the wires. The electron microscope Sirion NC 400 with an energy-dispersive X-ray (EDX) detector was used to perform the semi-quantitative chemical microanalysis. The sample preparation for the EDX on the longitudinal section of wire was carried out in following way: 1) wire samples were cold mounted using the compound Varidur 200; 2) the samples were ground with SiC paper; P2500, and P4000; 3) the samples were polished with Chemomet polishing cloth with sized alumina polishing paste 0.05 μm; 4) the samples were cleaned in an ultra-sound appliance and, finally; 5) the samples were positioned in an aluminium holder with a graphite strip and inserted into the chamber of the microscope. The EDX analyses were made on 12 different sites on each wire (longitudinal cross-section and on the surface of the wire).

2.2 Phase-transformation temperature

The important temperatures of the phase transformations in the NiTi wires are M_s (martensite – start) and M_f (martensite – finish) as well as A_s (austenite – start) and A_f (austenite – finish). In some SMA materials an intermediate R-phase could be formatted with a rhombohedral crystal structure. The start of the phase formatting is known as R_s and the end as R_f[14–16].

Under stress-free conditions these temperatures are commonly measured using DSC (differential scanning calorimetry). In our experiments the specimens for the DSC analyses were cut from orthodontic wires. Each specimen for the DSC analyses consisted of approximately long piece of wire 3–4 mm. The DSC analyses were conducted on a STA 449 NETZSCH machine by using a liquid-nitrogen cooling accessory. The specimen was heated to 100 °C and then cooled to –100 °C to obtain the cooling DSC curve. After that, the specimen was heated from –100 °C back to 100 °C to obtain the heating DSC curve. The cooling rate was 10 °C/min, as normally used with SMA NiTi.

2.3 Stress-Strain Curve

NiTi alloys have specific mechanical properties. The stress-strain curve from a static tensile test depends on microstructure (austenite or martensite).[17,18] The tensile tests were performed using a Zwick/Roell ZO 10 machine (**Figure 3**) under the following conditions:

- ambient temperature of 22 °C,
- strain rate of 0.025 s^{-1} (deformation velocity v = 1.5 mm/min),
- pre-load of 5 N.

The results are described in terms of the stress-strain (σ–ε) curve. The parameters considered as significant in describing the material behaviour of the curve are as follows: E_1 is the slope of the initial part of the loading curve (elastic modulus of austenite). E_2 is the slope of the transformation phase or region (austenite to martensite). E_3 is the slope of the final part of the curve (elastic modulus of austenite). σ_{SM} is the initial stress value of the transformation region or plateau. σ_{FM} is the final stress value of the transformation plateau. σ_f is the stress value at failure. ε_{SM} is the initial strain value of the transformation region or plateau. ε_{FM} is the final stress strain of the transformation plateau. ε_f is the strain value at failure.

One of most valuable properties of an ideal orthodontic archwire is good spring-back. This spring-back is the extent to which the activated wire is able to recover upon deactivation. In other words, wire may be deformed into loops or bends in complicated shapes employed to maximize their stored elastic energy and then return to their initial shape after deactivation.[2,19] Although SMAs can recover to their original austenitic phase, transformation and martensitic regions (up to the yield point of the material, where plastic deformation starts to fracture), orthodontic wires are almost never stressed in the martensitic region for orthodontic wire is just an important part, as shown in **Figure 2**[20].

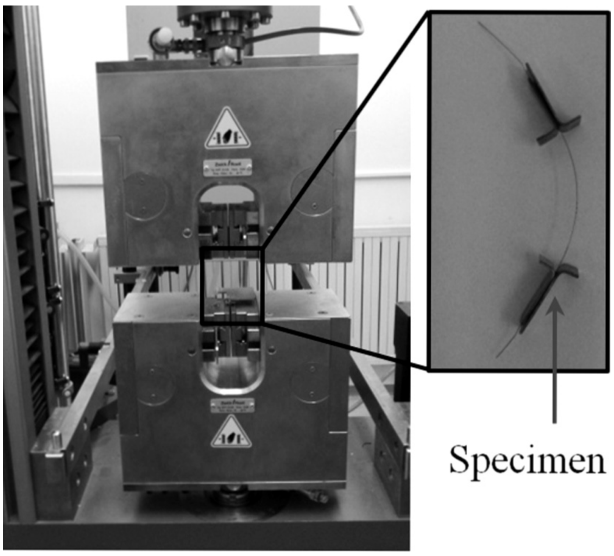

Figure 3: Tensile test for orthodontic wire with typical specimen
Slika 3: Natezni preizkus za ortodontsko žico z značilnim preizkušancem

3 RESULTS

3.1 EDX semi-quantitative chemical analysis

The average results of the EDX analyses are shown in **Table 2**, separately for the concentration of nickel and titanium in the longitudinal section (bulk material) and on the surface. A higher atomic fraction of nickel was found in the longitudinal section in three orthodontic wires (1, 3, 5). Moreover, in orthodontic wire 1 the atomic fraction of titanium was higher than nickel. In this wire the element Cu is added. Consequently, this alloy, when under stress, has a gentle and longer passage into the transformation area. Moreover, in wires 1 and 5 a difference in the content of Ni in the longitudinal section and on the surface was found, with a lower value of Ni. The manufacturing processes for the fabrication of NiTi wires is different and this is probably the reason for such findings. The other wires did not show any noticeable differences (max. about mole fraction x = 0.6 %).

Over 50 % of Ni was found in all wires, except in wire 1, where the Ni content is reduced by the presence of Cu. The Ni content in percentages is similar on the surface and in bulk (the noticeable difference is only visible in wire 2).

Table 2: Chemical composition on surface layer and bulk material of several NiTi wires in mole fractions, x/%
Tabela 2: Kemijska sestava površine in osnove Ni-Ti žic v molskih deležih, x/%

Orthodontic wire	Surface Layer			Bulk Material (longitudinal cross-section)		
	x(Ni)/% (± 0.1)	x(Ti)/% (± 0.1)	x(Cu)/% (± 0.1)	x(Ni)/% (± 0.1)	x(Ti)/% (± 0.1)	x(Cu)/% (± 0.1)
1	44.3	48.2	7.5	46.2	47.9	5.9
2	50.9	49.1	-	50.7	49.3	-
3	50.3	49.7	-	50.9	49.1	-
4	50.6	49.4	-	50.6	49.4	-
5	48.1	51.9	-	50.4	49.6	-
6	50.0	50.0	-	50.3	49.7	-

3.2 Phase-transformation temperature

Figure 4 presents the DSC plots for both the heating and cooling curves for separate orthodontic wires. All the curves on heating presented with temperatures A_f lower than 37 °C (body temperature), indicating an expression of the superelastic effect during an orthodontic treatment.

The temperatures of the phase transformation are presented in **Table 3**. The temperatures where there is full austenite stability are in the range from 14.3 °C to 26.8 °C. The R-phase appeared in the orthodontic wire 6. The temperatures where there is full martensite stability are in the range from –14.58 °C to –71.3 °C. Orthodontic wires 1, 4 and 6 begin their transition into the R phase or, respectively, into martensite, above 0 °C, while the temperature of the food consumed has the final influence

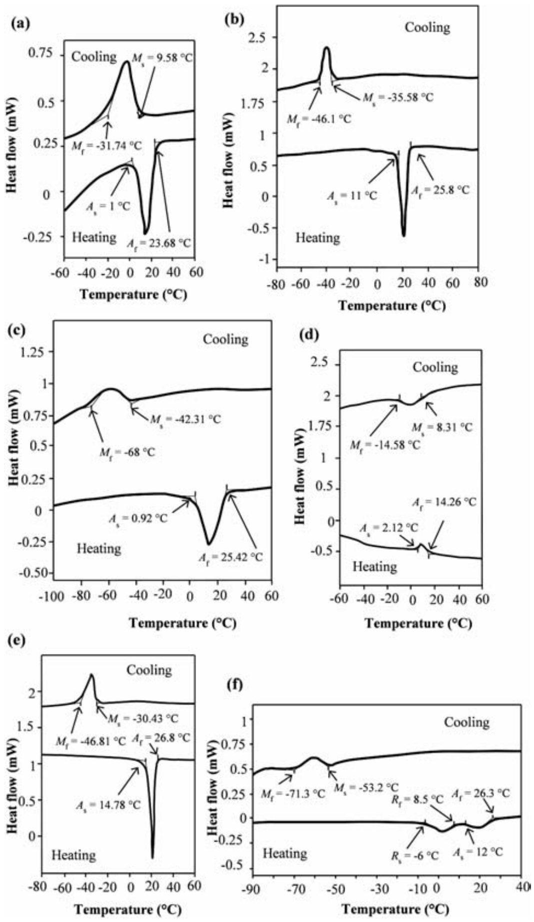

Figure 4: DSC curves and phase-transition temperatures for the orthodontic wire: a) orthodontic wire 1, b) orthodontic wire 2, c) orthodontic wire 3, d) orthodontic wire 4, e) orthodontic wire 5, f) orthodontic wire 6

Slika 4: DSC-krivulja in temperature faznih prehodov za ortodontsko žico: a) ortodontska žica 1, b) ortodontska žica 2, c) ortodontska žica 3, d) ortodontska žica 4, e) ortodontska žica 5, f) ortodontska žica 6

on the phase changes. This leads to changes of the SMA properties. Namely, these alloys, in the martensitic phase, show a lower modulus of elasticity, expressing higher forces on the teeth. In contrast to this, the consumption of food that warmer than body temperature causes the phase to change back to austenite, which is a consequence of stress increasing the passage into the transformation area (in accordance with the increasing temperature).

Table 3: Temperatures of phase transformation
Tabela 3: Temperature faznih prehodov

Orthodontic wire	M_f/°C	M_s/°C	R_s/°C	R_f/°C	A_s/°C	A_f/°C
1	–31.74	9.58	-	-	1.00	23.68
2	–46.10	–35.58	-	-	11.00	25.80
3	–68.00	–42.31	-	-	0.92	25.42
4	–14.58	8.31	-	-	2.12	14.26
5	–46.81	–30.43	-	-	14.78	26.80
6	–71.30	–53.20	–6.00	8.50	12.00	26.30

3.3 Stress-Strain Curve

Figure 5 shows the stress strain curve for each orthodontic wire. The results are presented in **Table 4**.

In **Figure 6a** the modulus of elasticity for the austenite, transformation and martensite phase is presented. The orthodontic wire 4 has the largest modulus of elasticity in the austenitic phase. The modulus of elasticity in the transformation phase of all the orthodontic wires is approximately at the same level varying from 3800 MPa to 6800 MPa. In an orthodontic treatment the desirable mechanical properties of the wires are changeable with regard to the treatment level. At the beginning of the treatment a low stiffness is important. At the end of orthodontic treatment, when the levelling stage is over and extensive tooth movement is needed, the stiffness of

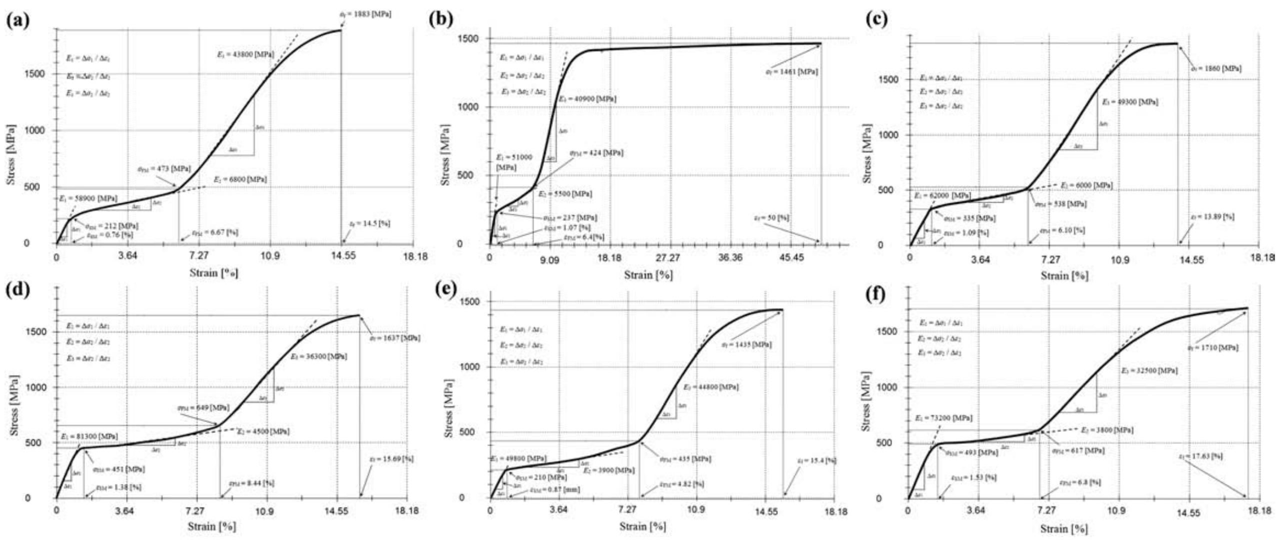

Figure 5: Characteristic parameters to describe material response, for a static-tensile test up to failure for the orthodontic wire: a) orthodontic wire 1, b) orthodontic wire 2, c) orthodontic wire 3, d) orthodontic wire 4, e) orthodontic wire 5, f) orthodontic wire 6

Slika 5: Značilni parametri za opis materialnega odziva pri nateznem preizkusu do porušitve ortodontske žice: a) ortodontska žica 1, b) ortodontska žica 2, c) ortodontska žica 3, d) ortodontska žica 4, e) ortodontska žica 5, f) ortodontska žica 6

Table 4: Comparison of mechanical properties of different, commercially available orthodontic wires during tensile test up to failure
Tabela 4: Primerjava mehanskih lastnosti različnih komercialno dostopnih ortodontskih žic med nateznim preizkusom do porušitve

Orthodontic wire	E_1/MPa	E_2/MPa	E_3/MPa	σ_{SM}/MPa	σ_{FM}/MPa	σ_f/MPa	ε_{SM}/%	ε_{FM}/%	ε_f/%
1	58900	6800	43800	212	473	1883	0.76	6.27	14.5
2	51000	5500	40900	237	424	1461	1.07	6.4	50
3	62000	6000	49300	335	538	1860	1.09	6.10	13.89
4	81300	4500	36300	451	649	1637	1.38	8.44	15.69
5	49800	3900	44800	210	435	1435	0.87	7.82	15.4
6	73200	3800	32500	493	617	1710	1.53	6.8	17.63

the wire must be higher. For this reason, it is clear that the optimal mechanical properties of orthodontic wires are different during the course of an orthodontic treatment.

It is important for orthodontic wires to have a low modulus of elasticity in the initial stage of treatment, when low forces are needed. This is one of the reasons why SMA NiTi wires are used in the initial stage of an orthodontic treatment.

Another important factor in orthodontic treatment is to ensure the application of continuous forces. The SMA NiTi provides nearly continuous force in the transformations plateau. **Figure 6b** shows the initial strain value for the transformation ε_{SM} and the value of the final strain ε_{FM} of the transformation. The wires 4 and 5 show a wide range of transformation plateau or strain and a good spring-back.

Figure 6c shows a comparison of the initial stress value of the transformation plateau σ_{SM} and the final stress value of the transformation plateau σ_{FM}. The orthodontic wire 6 shows the largest stresses. The orthodontic wire 1 shows the lowest stress σ_{SM}.

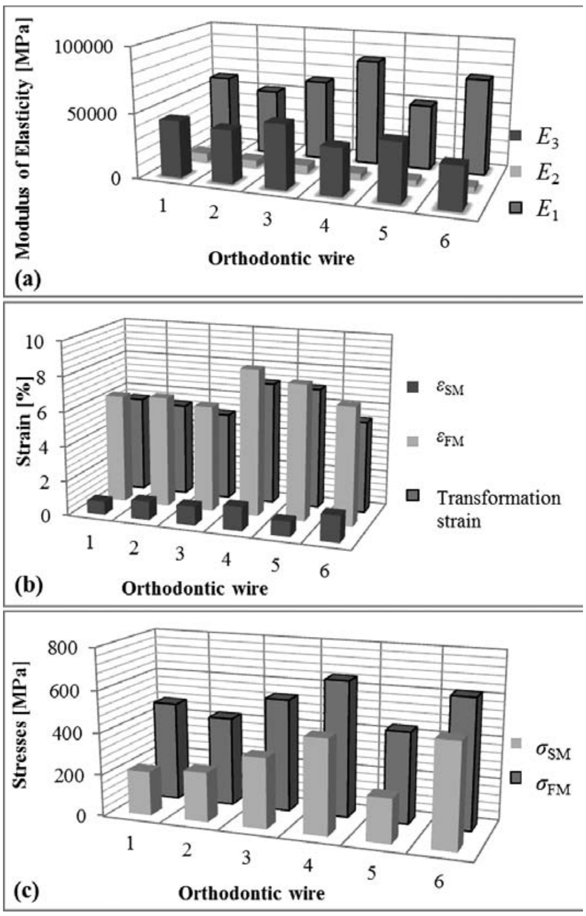

Figure 6: Comparison of orthodontic wires: a) modulus of elasticity for austenite E_1, transformation E_2 and martensitic E_3 phase; b) strain ε_{SM}, ε_{FM} and total transformation strain; c) experimental results for stress σ_{SM} and σ_{FM}
Slika 6: Primerjava ortodontskih žic: a) modul elastičnosti za avstenitno E_1, transformacijsko E_2 in martenzitno E_3 fazo; b) raztezek ε_{SM}, ε_{FM} in celotni transformacijski raztezek; c) eksperimentalni rezultati za napetost σ_{SM} and σ_{FM}

4 DISCUSSION

NiTi orthodontic wires have a complex metallurgical nature. The results of this paper show a correlation between the chemical composition, the phase transformation and the stress-strain curve of NiTi archwires used in orthodontic treatment. The investigation of the chemical composition showed a typical (equiatomic) composition for the nickel and the titanium in the SMA NiTi. The analysis of the phase-transformation temperatures of all six orthodontic wires showed that the temperature A_f, when the austenite phase is present, were below body temperature. All the wires exhibited a superelastic effect when they were used in the mouth. During the tensile test, the wires showed different elastic modulus, different slopes in the transformation phase and different martensite modulus. All of the wires showed the typical transformation plateau for SMA. Orthodontic wire 4 showed the maximum transformation plateau or strain (7.06 %). Whereas orthodontic wire 6 showed the least modulus of transformation plateau (E = 3800 MPa), which indicated the application of the lowest force on the teeth during orthodontic treatment.

The modulus of elasticity for the investigated wires was in the range between 49900 MPa and 81300 MPa in the austenitic phase, while in the transformation phase their values were between 3800 MPa and 6800 MPa. The modulus of elasticity for martensite phase are in the range between 40900 MPa and 49300 MPa.

In NiTi SMAs the influence of the chemical composition of the alloy is very important for the transformation temperatures and the connection between the transformation temperatures and the mechanical properties. Namely, the transformation temperature in the NiTi SMA depends on the content of nickel and titanium: increasing the nickel content decreases the A_f temperature. The values of A_f temperature in the NiTi

SMA change from 120 °C at 50 % Ni down to –40 °C at 51 % Ni[3]. A small change in the nickel content alters the A_f transformation temperature, above at which the SMA exhibits a superelastic behaviour.

The A_f temperature in orthodontic wire should be as similar as possible to body temperature (37 °C), namely, the SMA at this temperature shows a minimum stress (σ_{SM}) for the start of martensite transformation for the superelastic behaviour (wire 5 had the smallest difference between A_f and body temperature and minimum stress σ_{SM}). Decreasing the A_f temperature according to the body temperature increases the stress at which the SMA enters into the transformational plateau (wire 4 has the largest difference between A_f and body temperature and shows a large stress σ_{SM}).

In order to approach the necessary A_f temperature in the SMA for orthodontic wires it is necessary to have an accurate amount of nickel and titanium (x = 50.3 % up to 50.9 % Ni). Our uniaxial tensile tests were performed at ambient temperature (22 °C). The values for stress (σ_{SM}, σ_{FM}) increase with body temperature, while the modulus of elasticity stays unchanged.

The transformation strain decreases with temperature growing from the A_f temperature. Therefore, these are the greatest at the A_f temperature (wire 4 has a transformation strain of 7.06 % at room temperature 8 °C above the A_f temperature).

While the A_f temperatures of the other wires are higher than the temperature at which the tensile test has been performed, the transformation-induced strains are slightly different from those at body temperature. In **Table 4** we have listed the stress and strain at the rupture of the material; however, in orthodontic practice, such high stresses and strains are never achieved with the deformation of wires.

The rupture of the orthodontic wires is caused by the sudden changes of temperature in the mouth and, consequently, increasing stresses in the wire. The rapid changes of temperature in the mouth are provoked due to the ingestion of hot or cold foods or drinks. These temperatures can range from 2 °C to 60 °C. If the actual temperatures are below A_f, the stresses at which the martensitic transformation occurs are reduced and, therefore, the forces on the teeth are smaller. In contrast to this, at elevated temperatures (60 °C) the stress for the martensitic transformation (σ_{SM}) increases, which can lead to increased forces on the teeth. Temperature shocks in the mouth cannot be avoided, since they are part of everyday life in humans. In the orthodontic NiTi SMA wire it is important to minimize these shocks so that the wire is at its most effective to perform its function. For this, the mechanical properties, especially the superelasticity, are the most important.

5 CONCLUSIONS

In this work, for the first time, a combination of EDX, DSC and tensile-test analyses were used to determine the correlation between the chemical composition, the phase temperatures and the stress-strain curves of different orthodontic wires. The result of this investigation are reflected in a determination of the functional property (super-elasticity), which is the most important property of orthodontic wires. Based on the obtained results, it could be concluded that such a complex review is especially useful for the users of wires (i.e., orthodontists), because it reduces the chances of making a decision about the wrong wire material for special orthodontics problems.

A connection between clinical dentistry and a knowledge of materials science is necessary in order to achieve a better understanding of the orthodontic wire's behaviour.

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