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Mechanical testing of orthodontic archwires

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Abstract

Mechanical properties of orthodontic archwires are studied in this work in the micrometric domain. Measurements are performed on nickel-titanium archwires without and with different coatings as well as subjected for four weeks to an artificial saliva solution with different pH values (4.8 and 6.6). Since surface properties can have a meaningful influence on friction, corrosion or plaque accumulation, surface texture of the wires is measured by employing atomic force microscopy. On the other hand, with the aim of determining Young's modulus and the hardness, nanoindentation tests are performed for different peak load values. It is hence established that there is no statistically relevant deterioration of the surfaces after their exposure to saliva. Rh coating leads, in turn, to an increase of surface roughness. Young's moduli and hardness values tend to increase with increasing indentation depths, while they are not meaningfully affected by the coating or the corrosion in saliva.

Nickel-titanium archwires, coating and corrosion, AFM and nanoindentation testing, surface roughness, hardness, Young's modulus

1. Introduction

Orthodontic archwires with submillimetre cross-sections, used to correct anomalies in teeth position, must be aesthetically pleasing and biostable but also characterised by low friction, formability as well as high compliance. Mechanical properties of typically used archwire materials, such as their Young's modulus, hardness and especially surface texture, must thus be studied as they influence archwires' efficient use [1-2]. Three types of 0.02 inch x 0.02 inch archwires are hence studied:

- a superelastic nickel-titanium alloy with an untreated surface (NiTi, i.e. Nitinol known as a shape memory alloy): has good mechanical and clinical properties, sold as Sentalloy;
- NiTi archwire with an ion-implanted rhodium (Rh) coating: Rh NiTi sold as High Aesthetic;
- and the nitrified NiTi archwire: N NiTi sold as IonGuard.

To measure the relevant mechanical properties of archwire specimens, the equipment of the Precision Engineering Laboratory of the University of Rijeka, Croatia, is used. The surface texture of the samples is hence measured by employing the atomic force microscopy (AFM) mode of Bruker's Dimension Icon SPM, whereas Young's moduli and the hardness are determined by using a Keysight G200 Nanoindenter. Conclusions on the influence of the used coatings and of the corrosion on the measured mechanical properties of archwires are thus drawn.

2. Materials and methods

Specimens of each wire type are cut from arch forms. They are then subjected to an artificial saliva solution (1.5 g/L KCl, 1.5 g/L NaHCO₃, 0.5 g/L NaH₂PO₄xH₂O, 0.5 g/L KSCN, 0.9 g/L lactic acid) for four weeks at 37 °C. To simulate intraoral variations, two solutions with different pH values of artificial saliva (4.8 and 6.6) are adjusted with lactic acid and NaOH. Depending on the coating and the pH value, 6 different wire types are hence used in tests made at 23 °C.

AFM is considered as an appropriate tool for measuring surface topography of orthodontic wires, since it allows a high-resolution qualitative characterisation. In fact, surface properties, and in particular roughness, can have a meaningful influence not only on friction, but also on corrosion, adhesion, plaque accumulation and aesthetics of the archwires [1, 3]. Bruker's SPM apparatus is hence used in this work to perform two contact-mode AFM measurements on four samples of each considered wire type (affixed onto a sample holder – see Fig. 1 left). In total, 48 measurements are hence performed on 30 μm x 30 μm surfaces. The measurements are controlled via the instrument's NanoScope software that is also used to flatten measurement data to filter the inclination of the probe with respect to the surface of the sample. The used probe is Bruker's SNL-10 highresolution probe with a 2 nm Si tip radius mounted on a 0.6 μm thick triangular Si_3N_4 cantilever with a bending stiffness of 0.12 N/m. Surface topographies, and in particular the values of the arithmetic average surface roughness Ra, the RMS roughness R_q and the maximum height R_Z , are hence obtained.



Figure 1. Archwires mounted on the AFM sample holder (left) and specimens mounted into the Keysight G200 Nanoindenter (right).

Nanoindentation is an accurate way to probe the mechanical characteristics of the surface layers of orthodontic archwires [3]. In fact, the used Keysight device can generate forces with a resolution of 50 nN and measure displacements with a resolution of 0.01 nm. The indentation hardness H_{IT} and indentation Young's modulus E_{IT} measurements are hence performed in accordance to ISO 14577 by using a 20 nm Berkovich tip; nanoindenter frame compliance and tip sharpness calibration are performed on a standardised fused-Si reference sample. A 10 s time to load, a peak hold time of 1 s and allowable drift rates

of 0.2 nm/s are set via instrument's software that is used also to acquire measurement data. Four wires of the same type are hence bonded to a sample holder with a cyanoacrylate adhesive, while three holders are positioned and bonded onto a standard Al sample mount, as this solution has proven to be the most stable and insensitive to perturbations. Two sample mounts, each with 12 wires, are hence carefully aligned in their seats to guarantee a flat mounting of all the samples (Fig. 1 right). Since in literature it is evidenced that E_{IT} and H_{IT} vary depending on loading conditions [3], each of the 24 archwires is hence indented in a 4 x 4 pattern of points with a peak load of 20 mN (corresponding to an average indentation depth of ~ 1 μ m) and then, in a different area, with a 100 mN peak force (indentation depth of ~ 2 μ m). More than 750 indents are thus performed.

3. Results and discussion

Obtained representative 3D AFM images of the surfaces of the analysed archwires are shown in Fig. 2 whereas the R_a , R_q and R_Z values with the respective standard deviations are reported in table 1 for each of the 6 wire type – pH value combinations. It can thus be inferred that, contrary to what was reported in [2], there is no significant correlation between corrosion in saliva with variable pH values and surface roughness. It is clear, however that, in accordance also with [1-2], the Rh coating tends to increase the roughness. The values of the characteristic parameters for the Rh NiTi archwires ($R_a \approx 155 \text{ nm}$, $R_a \approx 205 \text{ nm}$, $R_Z \approx 1770$ nm) are thus higher than those of the other two considered wire types, which are, in turn, approximately $R_q \approx 140 \text{ nm},$ equivalent $(R_a \approx 110 \text{ nm},$ $R_Z \approx 1150 \text{ nm}$). Roughness of the plain NiTi archwire tends, however, to be higher than that reported in previous literature [1-2], while, in general, the dispersion of data, related to optically visible material inhomogeneity that could be related to the differences in the production processes, is rather large.



Figure 2. Topographies of NiTi (~ N NiTi) and Rh NiTi wires with pH 6.6.

	Table 1 Surface roughness	parameters of the a	nalysed archwires.
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	рΗ	R _a /nm	R _a /nm	R _z /nm
NiTi	4.8	98.5 ± 14.8	125.1 ± 18.3	1175 ± 389
	6.6	120.3 ± 41.7	151.8 ± 47.4	1147 ± 400
Rh	4.8	162.4 ± 40.7	211.4 ± 50.8	1757 ± 521
NiTi	6.6	152.8 ± 22.8	199.9 ± 30.2	1784 ± 771
N	4.8	117.6 ± 21.7	150.8 ± 27.8	1614 ± 1023
NiTi	6.6	109.0 ± 12.4	134.8 ± 15.6	1023 ± 237

A typical diagram of the performed nanoindentations is shown in Fig. 3, while the obtained E_{IT} and H_{IT} values, with the respective standard deviations, are given in table 2. It can hence be noted that there is seemingly no deterioration of the mechanical properties of the archwires with their exposure to saliva. E_{IT} and H_{IT} values are clearly lower (roughly 30 % in the case of E_{IT} and up to 3-6 times in the case of H_{IT}) than those reported in [3]. In that case, however, not only the influence of the pH value was not considered, but the surfaces of the samples were also ground prior to nanoindentation, which clearly influences surface properties by levelling the present irregularities and inhomogeneity. Also, the herein obtained E_{IT} and H_{IT} values tend to increase by rising the peak load (indentation depth), which is in contrast with the results obtained in [3] and could be related to the difference in the products evaluated in the two cases. The values of H_{IT} and E_{IT} obtained in this work do not correlate with the variation of surface coating either. Moreover, as in the case of surface roughness, there is a large dispersion of measurement values, confirming material inhomogeneity.



Figure 3. Typical nanoindentation curves for the considered archwires.

Table 2 Archwires' indentation Young's modulus E_{IT} and hardness H_{IT}.

		<i>E_{IN}/</i> GPa		H _{IN} /GPa	
	рп	20 mN	100 mN	20 mN	100 mN
NiTi	4.8	45.5 ± 12.2	52.1 ± 9.8	0.8 ± 0.4	1.3 ± 0.5
	6.6	44.7 ± 10.3	50.1 ± 26.4	0.9 ±0.4	2.0 ± 1.4
Rh	4.8	31.4 ± 18.3	48.1 ± 14.5	0.6 ± 0.8	1.1 ± 0.7
NiTi	6.6	42.7 ± 13.4	43.9 ± 21.1	0.7 ± 0.4	1.5 ± 0.5
Ν	4.8	33.8 ± 12.1	40.3 ± 14.1	0.5 ± 0.4	1.0 ± 0.3
NiTi	6.6	52.1 ± 15.7	41.7 ± 9.6	1.9 ± 1.1	1.3 ± 0.6

4. Conclusions and outlook

A thorough analysis of the surface properties of orthodontic archwires with different coatings and pH values is performed in this work on the microscale by using AFM and nanoindentation. It is established that there is no deterioration of the markedly inhomogeneous surfaces with their exposure to saliva, regardless of its level of acidity. The surface quality is, in turn, related to non-standardised production and coating processes of the wires. Confirming prior art, it is established that Rh coating leads to an increase of surface roughness that could negatively affect friction, corrosion and plaque formation. On the other hand, the E_{IT} and H_{IT} values of archwires' surfaces are not affected by varying coating and corrosion caused by the acidulated (pH 4.8) and neutral (pH 6.6) saliva, whereas the obtained values are different from those reported in literature. This is related to the fact that in prior art surfaces of the samples were ground before measurements, which levels surface irregularities. E_{IT} and H_{IT} tend to increase with rising loads/depths of indentation.

In future work, the herein presented data will be correlated to tests on friction forces and other mechanical properties performed on the same specimens. Recently introduced diamondlike carbon coated archwires will also be considered, since they should imply a decrease of friction while significantly increasing hardness. An attempt will also be made to correlate the measured mechanical properties to information about the production process of the archwires, including information on the deposition processes of the coating materials.

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Essential bibliography

- [1] D'Antò V et al. 2012 Angle Orthodontists 82/5 922-8
- [2] Rongo R et al. 2014 Angle Orthodontists 84/4 665-72
- [3] lijima M, Muguruma T, Brantley W A and Mizoguchi I 2011 Am. J. Orthodontics & Dentofacial Orthopedics **140**/1 65-71