Effects of Indentation Load on the Mechanical Behavior of Orthodontic Wire Alloys by Dynamic Micro-Indentation Method

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Abstract

Selecting the appropriate wire alloys during orthodontic treatment requires knowledge of the biomechanical and clinical applications of various metallic orthodontic wires. Micro-indentation hardness testing is a convenient means of investigating the mechanical properties of a small volume of materials. The aim of this study was to apply the dynamic micro-indentation method as a simple method for determining the mechanical behavior of orthodontic wire alloys. Moreover, through this method, the effect of the indentation peak load on the mechanical behavior of orthodontic wire alloys was investigated. Four commercially available orthodontic wire alloys (stainless steel, cobalt-chromium-nickel alloy, s-titanium alloy and nickel-titanium alloy) were tested. All specimens were straight and round with a diameter of 0.018-inch. Dynamic hardness and elastic modulus of the orthodontic wire alloys under peak loads of 50, 100 and 1,000 mN were determined using a dynamic ultra-micro-hardness tester fitted with a Berkovich indenter tip. The elastic recovery on the nickel-titanium alloy was higher than on the others on unloading of micro-indentation load and penetration depth curves. Moreover, for all orthodontic wire alloys, both dynamic hardness and elastic modulus decreased with increasing indentation peak load. Thus, we confirmed that the hardness and elastic modulus of orthodontic wire alloys on micro indentation scales is dependent on the indentation size effect. In conclusion, the dynamic micro-indentation method is a useful technique for determining mechanical properties within very small areas on the surface of orthodontic wires without complex sample preparation.

Keywords :

orthodontic wire alloys; dynamic micro-indentation method; dynamic hardness; elastic modulus; indentation peak load

Introduction

Orthodontic wires made from different alloys are widely used as orthodontic appliances during all phases of orthodontic treatment. Among available orthodontic wire alloys, stainless steel, cobalt-chromium-nickel alloy, β -titanium alloy and nickel-titanium alloy are the most commonly used (1, 2). Based on the mechanical properties of these orthodontic wire alloys, it is possible to match phases of treatment with orthodontic wires. Therefore, knowledge of the mechanical behavior of various orthodontic wire alloys is important for optimal wire selection.

With regard to mechanical properties, indentation hardness tests are the most commonly used non-destructive testing procedures in the metal industry and research because they provide an easy, inexpensive and reliable method for evaluating basic properties of materials (3, 4). The indentation hardness of materials is measured by forcing an indenter having specific geometry, such as a ball, cone or pyramid shape, into the specimen surface. Generally, there are two methods of indentation hardness measurement in use. The conventional procedure of hardness testing consists of applying a fixed load on a diamond indenter and measuring, with the help of optical microscopy, the dimensions of the resultant indentation on the surface of the test material after unloading. However, it is difficult to

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Table 1 Orthodontic wires tested

ID	Wire alloy	Composition (wt%) *	Manufacturer	Lot number
SS	Stainless steel	18–20% Cr, 8–10.5% Ni, 2% Mn maximum, 1% Si maximum, 0.08% C maximum, 0.045% P maximum, 0.03% S maximum, balance mainly Fe	Tomy, Tokyo, Japan	A2Z1
Co-Cr	Cobalt-chromium-nickel (Elgiloy Blue)	40% Co, 20% Cr, 15.81% Fe, 15% Ni, 7% Mo, 2% Mn, 0.15% C, 0.01% Be maximum	Rocky Mountain Orthodontics, Denver, CO, USA	WO-247890
TMA	β -titanium	10.0–13.0 % Mo, 4.5–7.5% Zr, 3.75–5.25% Sn, 5% (Fe, O, H, C, N ₂), balance mainly Ti	Ormco, Glendora, CA, USA	12F83
Ni-Ti	Nickel-titanium	55.0% Ni, 45.0% Ti	American Orthodontics, Sheboygan, WI, USA	060211W3

* Based on information from the manufacturer.

measure such dimensions optically with a high degree of accuracy, for indents on the micro-/nano-scale. On the other hand, the dynamic nano- or micro-indentation method offers great advantages over conventional hardness testing. In this depth-sensing indentation hardness test, a diamond indenter tip is pressed into the specimen to a given maximum load or depth, and is then removed. The load on, as well as the displacement of the indenter are subsequently recorded. This method can provide well-defined mechanical parameters such as the dynamic hardness and the elastic modulus. Accordingly, dynamic nano- or micro-indentation method is considered to be an effective tool for evaluating mechanical properties such as the dynamic hardness and elastic modulus of various orthodontic wire alloys. Iijima et al. (5) investigated the mechanical properties of available orthodontic wire alloys under low indentation loads ranging from 2 to 100 mN using the nano-indentation method. The authors indicated that dynamic hardness and elastic modulus decreased with increasing indentation peak load. To date, there have been a few reports on the mechanical properties of orthodontic wires by nano- or micro-indentation testing (5, 6). In particular, the effects of experimental parameters on mechanical behavior, such as the degree of plastic and elastic deformation, dynamic hardness and elastic modulus, obtained by the micro-indentation method are uncertain for orthodontic wire alloys.

In the present study, the dynamic micro-indentation method was applied in order to examine the mechanical behavior of orthodontic wire alloys. Moreover, the effects of indentation peak load on the degree of plastic and elastic deformation, dynamic hardness and elastic modulus of the

orthodontic wire alloys obtained by this method were investigated.

Materials and Methods

Four commercially available orthodontic wires (stainless steel, cobalt-chromium-nickel alloy, β -titanium alloy and nickel-titanium alloy) were evaluated. Table 1 lists the metallic orthodontic wires, their codes, composition and other information. All specimens were straight and round with a diameter of 0.018-inch. Ten specimens of 30 mm in length were prepared for testing. Investigation of the mechanical behavior of orthodontic wire alloys was performed using a dynamic ultra-micro-hardness tester (DUH-211; Shimadzu Co., Kyoto, Japan; Fig. 1a) fitted with a Berkovich indenter tip (Fig. 1b). Specimens were fixed onto the attached holder. Dynamic micro-indentation primarily consists of a controlled load (P) applied through a diamond tip that is in contact with a smooth surface. Penetration depth (h) of indentation is continuously recorded as a function of load. Fig. 2 shows a micrograph of the typical indented specimen surface and a schematic illustration of the typical micro-indentation load-penetration depth curve obtained from dynamic micro-indentation tests. During indenter loading and unloading, the specimen is subjected to both plastic deformation (h_p) and elastic deformation (h_e). The total deformation (h_t) is the sum of h_p and h_e shown on the micro-indentation load versus penetration depth curve in Fig. 2(b). Moreover, dynamic hardness and elastic modulus can be obtained from the indentation load and penetration depth data (7).

Dynamic hardness (DH) of the sample was calculated

Fig.1 Dynamic ultra-micro-hardness testing system used in this study. (a) Photograph of dynamic ultra-micro-hardness tester. (b) Schematic image of the geometry of the Berkovich indenter tip. Tip height, L is defined as " $L = (1/3)(L_1 + L_2 + L_3)$ ".

using the following equation:

$$DH \equiv \alpha P / h^2 \quad (1)$$

where, α is a geometric constant of the Berkovich indenter (3.8584), P is the applied load during indentation test, and h is the penetration depth of indentation.

The elastic modulus (E) of the sample was calculated using the following equation:

$$1/E_r = (1 - V^2)/E + (1 - V_i^2)/E_i \quad (2)$$

where, E_r is the reduced elastic modulus from the indenter, V is the Poisson ratio for the sample, V_i is the Poisson ratio for the Berkovich indenter (0.07), and E_i is the modulus of the Berkovich indenter (1140 GPa).

In the present study, dynamic micro-indentation tests were carried out with peak loads (P_{max}), of 50, 100 and 1,000 mN in order to investigate the effect of indentation peak load on the dynamic hardness and elastic modulus of orthodontic wire alloys. The load rate was kept constant at 13.32 mN/s, and hold time at maximum load was set at 15 s. Dynamic micro-indentation results such as dynamic hard-

Fig.2 Indented surface and indentation load-penetration depth curve obtained from dynamic ultra-micro-hardness test. (a) Micrograph of typical indented specimen surface (Magnification $\times 50$). (b) Schematic illustration of typical micro-indentation load versus penetration depth curve.

ness and elastic modulus were obtained as mean values of ten measurements ($n = 10$). The results of dynamic hardness and elastic modulus were examined by analysis of variance and tested using the Student's multiple comparisons test among means at $p = 0.05$.

Results

Typical indentation load-penetration depth curves of orthodontic wire alloys with indentation peak loads of 10, 50 and 1,000 mN are shown in Fig.3. For all orthodontic wire alloy specimens, penetration depth increased with indentation peak load. In addition, it was confirmed that indentation load-penetration depth curves under different peak loads

Fig.3 Typical load-indentation depth curves for four orthodontic wires obtained from dynamic ultra-micro-hardness test.

Table2 Total, plastic and elastic deformations of orthodontic wires at different indentation peak loads

ID	Indentation peak load, $P_{\max}$ (mN)	Total deformation, h_t (μm)	Plastic deformation, h_p (μm)	h_p/h_t (%)	Elastic deformation, h_e (μm)	h_e/h_t (%)
SS	50	0.709 ± 0.034	0.499 ± 0.036	70.3	0.210 ± 0.022	29.7
	100	1.048 ± 0.040	0.757 ± 0.032	72.3	0.291 ± 0.028	27.7
	1,000	3.869 ± 0.167	2.670 ± 0.118	69.0	1.199 ± 0.121	31.0
Co-Cr	50	0.659 ± 0.024	0.484 ± 0.030	73.4	0.175 ± 0.014	26.6
	100	1.014 ± 0.033	0.761 ± 0.022	75.1	0.253 ± 0.024	24.9
	1,000	4.277 ± 1.404	3.304 ± 1.412	77.2	0.973 ± 0.111	22.8
TMA	50	0.867 ± 0.083	0.567 ± 0.091	65.5	0.299 ± 0.034	34.5
	100	1.287 ± 0.148	0.845 ± 0.148	65.7	0.442 ± 0.048	34.3
	1,000	4.929 ± 0.300	3.164 ± 0.234	64.2	1.765 ± 0.202	35.8
Ni-Ti	50	0.968 ± 0.088	0.430 ± 0.063	44.4	0.538 ± 0.046	55.6
	100	1.406 ± 0.119	0.626 ± 0.061	44.6	0.780 ± 0.084	55.4
	1,000	5.074 ± 0.394	2.250 ± 0.135	44.3	2.824 ± 0.349	55.7

Fig.4 Dynamic hardness of the four orthodontic wires. Values connected by horizontal bars with '*' are not significantly different from one another ($p > 0.05$).

Fig.5 Elastic moduli of the four orthodontic wires. Values connected by horizontal bars with '*' are not significantly different from one another ($p > 0.05$).

can be accurately fitted to one curve due to their overlapping characteristics. Moreover, Ni-Ti showed the highest elastic recovery on orthodontic wire alloys under unloading. Table 2 shows the total deformation (h_t) values, which are the sum of plastic deformation (h_p) and elastic deformation (h_e), under indentation peak loads of 50, 100 and 1,000 mN. Here, indentation deformation $h_t = h_p + h_e$ is always satisfied. The h_p/h_t ratio and h_e/h_t ratio are also listed. It was confirmed that all types of orthodontic wire alloy have similar h_e/h_t (or h_p/h_t) ratios through different peak loads; the h_e/h_t ratio ranges of SS, Co-Cr, TMA and Ni-Ti were 27.7–31.0, 22.8–26.6, 34.3–35.8 and 55.4–55.7%, respectively, at indentation peak loads between 50 and 1,000 mN. In addition, it was apparent that the Ni-Ti has higher h_e/h_t ratios representing a higher degree of elastic recovery in comparison with other specimens. Fig. 4 shows the dynamic hardness of four orthodontic wire alloys at different indentation peak loads. At a peak load of 50 mN, the dynamic hardness of SS, Co-Cr, TMA and Ni-Ti was 397.9 ± 38.4 , 459.9 ± 34.2 , 271.5 ± 58.7 and 217.1 ± 34.0 , respectively. When the peak load was increased from 50 to 1,000 mN, the dynamic hardness of SS, Co-Cr, TMA and Ni-Ti decreased to 261.6 ± 22.0 , 256.9 ± 98.9 , 162.0 ± 20.0 and 153.5 ± 21.7 , respectively. Fig. 5 shows elastic moduli of the four orthodontic wire alloys at different indentation peak loads. At a peak load of 50 mN, the elastic moduli of SS, Co-Cr, TMA and Ni-Ti were 143.2 ± 18.3 , 179.0 ± 17.3 , 78.0 ± 12.0 and 45.9 ± 8.7 GPa, respectively. When the peak load was increased from 50 to 1,000 mN, the elastic moduli of SS, Co-Cr, TMA and Ni-Ti decreased to 86.2 ± 9.4 , 99.6

± 26.9 , 46.0 ± 6.2 and 34.1 ± 4.6 , respectively. Therefore, both dynamic hardness and elastic modulus decreased with increasing indentation peak load.

Discussion

Orthodontic wire alloys require proper characterization to predict their outcome when used clinically. The mechanical properties generally vary across the same types of wire alloy (8). For example, the Elgiloy used as a cobalt-chromium-nickel orthodontic wire alloy in the present study is available in four tempers (levels of resilience) that are color-coded by the manufacturer: Blue (soft); Yellow (ductile); Green (semi-resilient); and Red (resilient). Discussions with the manufacturer indicate that all four alloy tempers have the same composition; differences in mechanical properties arise from variations in wire processing (9). Therefore, a simple approach for determining the mechanical properties of orthodontic wires is required. Micro-indentation is a depth sensing technique that can accurately characterize the mechanical properties of almost all types of solid materials on a small scale. This method is able to continuously record the penetration depth of the specimen during dynamic loading and unloading at the indentation tip while providing information on the degree of plastic and elastic deformation, the hardness and elastic modulus of the material. Knowledge of these mechanical properties for orthodontic wires is very helpful to the clinician in the design and application of optimal force systems during orthodontic treatment. In the present study, the micro-indentation method was applied to investigate

mechanical properties such as dynamic hardness and elastic modulus of four commercially available orthodontic wires at different indentation peak loads.

Firstly, the indentation load-penetration depth curves were obtained during indenter loading and unloading. For all samples, note that the indentation load-penetration depth curves for various indentation peak loads correspond very well with one another (Fig.3). This indicates the high accuracy and reproducibility of the indentation method for orthodontic wire. Furthermore, the load-depth curves clearly indicate that the elastic recovery on the Ni-Ti is higher when compared with the others. In addition, the h_e/h_t ratio of Ni-Ti was higher than that of other wire alloys (Table2). Numerous studies have been conducted on the existence of superelastic behavior in Ni-Ti wires obtained by flexural testing(10-12). Meanwhile, it is interesting that the high elastic recovery associated with the superelastic behavior on the surface of Ni-Ti wire was detected by load-depth curves obtained using a dynamic ultra-micro-hardness tester in the present study. This finding indicates that surface properties were linked to bulk characterization of orthodontic wire alloys. Thus, the mechanical behavior on the surface of orthodontic wires is useful for understanding the surface details of the product and the components' contribution to the overall surface properties of an orthodontic wire. Moreover, it can also be seen that for all specimens, the deformation rate is not equal to zero when the indentation peak load is maintained for a holding time of 15s. This is due to the creep behavior of the material(13), and there were no large differences in creep behavior among the samples.

Secondly, dynamic hardness and elastic modulus, which are the two essential mechanical properties of the four commercially available orthodontic wire alloys were calculated from indentation load-penetration depth data. As shown in Figs.4 and 5, the mechanical properties among the 4 commercially available orthodontic wire alloys showed the same rank order under varying indentation peak loads. Stainless steel and cobalt-chromium-nickel alloys are known to have similar high stiffness. In comparison with these alloys, nickel-titanium and β -titanium alloys have low stiffness. In the present study, the rank order of the mechanical properties of orthodontic wire alloys obtained using the dynamic micro-indentation method is in accord with those reported in previous studies(1, 9, 14), while both the dynamic hardness and elastic modulus decreased with

increasing indentation peak load. Hardness and modulus of materials should not vary with indentation load, as these are accepted as an inherent material property. However, there are several reports of the effect of indentation peak load on dynamic hardness and elastic modulus of materials(15-17). For example, Ho and Marcolongo (16) evaluated the interfacial mechanics of bioactive composites composed of polymethylmethacrylate and hydroxyapatite for mandibular bone substitutes, under indentation peak loads ranging from 1 to 100mN by the nano-indentation technique. The authors reported that the mechanical properties decreased with increasing peak load up to 8 mN, and these properties become relatively constant. Ozun et al. (17) investigated the dynamic hardness and elastic modulus of polycrystalline superconductors under indentation peak loads between 490 and 1,220mN using the micro-indentation technique, and also found that the dynamic hardness and elastic modulus decreased with increasing indentation peak load. Our experimental results agree with the phenomenon reported in these studies. This phenomenon is due to the indentation size effect (ISE), for which there is a decrease in hardness with increasing indentation size (or load) for a penetration depth range typically less than 10 μ m(18-20). Generally, it is known that the ISE is related to surface effects, strain gradient effects, structural non-uniformity of the deformed volume, changes in the contribution of elastic and plastic deformation at indentation, and friction between the indenter and the sample (19). With regard to orthodontic wires, the dependence of indentation load (range, 2-100 mN) on the hardness of orthodontic wires obtained by nano-indentation testing has been confirmed (5). In orthodontic treatment, tooth movements and the associated changes are the result of an applied force system, and the tissue response to it. Thus, it is assumed that an optimal force system is important for an adequate biological response. Meanwhile, a wide range of optimal forces (over 180mN) at the initial stage of orthodontic tooth movement have been reported in a review article(21). To obtain a satisfactory understanding of the mechanical behavior of orthodontic wires during orthodontic treatment, nano- and/or micro-indentation testing of orthodontic wire alloys must be undertaken over a wide range of indentation loads. In the present study, the mechanical properties of orthodontic wire alloys were therefore investigated under both high indentation loads and the low indentation loads applied by Iijima et al.(5). As a result, we confirmed that the dependence of indentation

peak load (range, 50–1,000mN) on the hardness of orthodontic wires. Thus, the existence of the ISE in orthodontic wire alloys was exhibited at indentation loads ranging from 2 to 1,000mN using nano- and micro-indentations. In other words, the values for the mechanical properties of orthodontic wire alloys obtained by applying the nano- and micro-indentation method are influenced by indentation load level, although the rank order of mechanical properties among the orthodontic wire alloys is the same under the various indentation loads. Therefore, it is essential that the mechanical properties of orthodontic wire alloys be measured at the same indentation load level, when comparing mechanical properties obtained by the nano- or micro-indentation test. More research is required to further examine the relationship between the experimental parameters and mechanical properties determined by the micro-indentation method.

Finally, it is important for orthodontists to have reliable information on the mechanical properties of commercially available orthodontic wires. The advantage of the dynamic micro-indentation test performed in the present study is the relative simplicity of the experimental setup, in comparison with other mechanical test procedures. In light of this finding, the dynamic micro-indentation test has proven to be an effective method for evaluating the mechanical behavior of orthodontic wires.

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