

Comparisons of nanoindentation, 3-point bending, and tension tests for orthodontic wires

Masahiro Iijima,^a Takeshi Muguruma,^b William A. Brantley,^c and Itaru Mizoguchi^d

Ishikari-Tobetsu, Japan, and Columbus, Ohio

Introduction: The purposes of this study were to obtain information about mechanical properties with the nanoindentation test for representative wire alloys and compare the results with conventional mechanical tests.

Methods: Archwires having 0.016×0.022 -in cross sections were obtained of 1 stainless steel, 1 cobalt-chromium-nickel, 1 beta-titanium alloy, and 2 nickel-titanium products. Specimens of as-received wires were subjected to nanoindentation testing along the external surfaces and over polished cross sections to obtain values of hardness and elastic modulus. Other specimens of as-received wires were subjected to Vickers hardness, 3-point bending, and tension tests. All testing was performed at 25°C. **Results:** Differences were found in hardness and elastic modulus obtained with the nanoindentation test at the external and cross-sectioned surfaces and with the conventional mechanical-property tests. Mechanical properties obtained with the nanoindentation test generally varied with indentation depth. **Conclusions:** The 3 testing methods did not yield identical values of hardness and elastic modulus, although the order among the 5 wire products was the same. Variations in results for the nanoindentation and conventional mechanical property tests can be attributed to the different material volumes sampled, different work-hardening levels, and an oxide layer on the wire surface. (Am J Orthod Dentofacial Orthop 2011;140:65-71)

Orthodontic wires, which generate the biomechanical forces communicated through brackets for tooth movement, are central to the practice of orthodontics. In the rational selection of wires for a particular treatment, an orthodontist should consider a variety of mechanical properties, such as elastic range or springback, elastic modulus, and yield strength.¹ Currently, orthodontists principally use wires of 4 major base metal alloy types: stainless steel, cobalt-chromium-nickel, nickel-titanium, and beta-titanium,^{1,2} and a variety of bending and tension tests have been performed to

determine elastic modulus and yield strength.³⁻⁷ In addition, the property of hardness has major importance in the comparison of dental materials,^{1,8} and microhardness testing has been used to characterize orthodontic wires.^{1,9} Hardness is influenced by the microstructure and is expected to be related to the yield strength for orthodontic wires of the same alloy type.¹⁰

Recent advances in the nanoindentation test have allowed the measurement of mechanical properties for extremely small volumes of materials where the contact radius is less than 100 nm.¹¹⁻¹⁷ In nanoindentation testing, the load and displacement are recorded with a high-resolution displacement gauge during the indentation process, and the area of the indentation is obtained from the geometric form of the indenter without having to actually observe the indentation; thereby, the hardness of the specimen can be calculated. In addition, the elastic modulus for this small volume of material can be obtained mathematically from the load-displacement curve by using suitable software. A recent study used the nanoindentation test to investigate the effects of decontamination and clinical exposure on the elastic modulus, hardness, and surface roughness of 2 frequently used orthodontic wires.¹⁸ However, little is known about the relationship between the mechanical properties (hardness and elastic modulus) obtained by the nanoindentation test and the conventional mechanical tests.

^aAssociate professor, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Ishikari-Tobetsu, Japan.

^bInstructor, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Ishikari-Tobetsu, Japan.

^cProfessor, Division of Restorative and Prosthetic Dentistry, College of Dentistry, Ohio State University, Columbus.

^dProfessor, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Ishikari-Tobetsu, Japan.

The authors report no commercial, proprietary, or financial interest in the product or companies described in this article.

Reprint requests to: Masahiro Iijima, Division of Orthodontics and Dentofacial Orthopedics, Department of Oral Growth and Development, School of Dentistry, Health Sciences University of Hokkaido, Kanazawa 1757, Ishikari-Tobetsu, Hokkaido 061-0293, Japan; e-mail, ijijima@hoku-iryo-u.ac.jp.

Submitted, July 2009; revised and accepted, November 2009.

0889-5406/\$36.00

Copyright © 2011 by the American Association of Orthodontists.

doi:10.1016/j.ajodo.2009.11.015

Table I. Summary of orthodontic wires used in this study

Product	Manufacturer	Lot	Alloy type
Copper Ni-Ti (A _r 35°C)	Ormco, Orange Calif	02C194C	Nickel- titanium- copper
Nitinol Classic	3M Unitek, Monrovia, Calif	376	Nickel- titanium
Resolve	Dentsply GAC International, Bohemia, NY	F0712092	Beta- titanium
Unitek Stainless Steel	3M Unitek, Monrovia, Calif	507	Austenitic stainless steel
Elgiloy Blue	Rocky Mountain Orthodontics, Denver, Colo	040308	Cobalt- chromium- nickel

The purposes of this study were to use the nanoindentation test to examine commercial orthodontic wires and to compare the mechanical properties (hardness and elastic modulus) with values of hardness and elastic modulus obtained by the conventional Vickers hardness test, 3-point bending test, and tension test.

MATERIAL AND METHODS

Five wire products—2 nickel-titanium, 1 beta-titanium, 1 stainless steel, and 1 cobalt-chromium-nickel, with cross-section dimensions of 0.016 × 0.022-in—were obtained from 4 manufacturers (Table I). The as-received wires were cut into 25-mm segments with a slow-speed, water-cooled diamond saw (Isomet 11-1280; Buehler, Lake Bluff, Ill) for nanoindentation, Vickers hardness measurements, and 3-point bending tests. All wires were obtained as preformed arches. The wires for tension testing were sectioned at the midline, and the most straight portions were used. The Elgiloy Blue wires (Rocky Mountain Orthodontics, Denver, Colo) were heat treated as suggested by the manufacturer by using an electrical resistance welding apparatus (SS-100; Sankin, Tokyo, Japan) before mechanical testing.

The as-received wires were fixed on the specimen stage with adhesive resin (Superbond Orthomite; Sun Medical, Shiga, Japan) for the nanoindentation test on the external surface. For nanoindentation testing of the cross sections, as-received wires were encapsulated in epoxy resin (Epofix; Struers, Copenhagen, Denmark), and the specimens were ground (600-grit sandpaper) and then polished by using a series of silicon-carbide abrasive papers and a final slurry of 0.05- μ m alumina particles. All nanoindentation testing (ENT-1100a; Elixion, Tokyo, Japan) was carried out at 25°C by using a Berkovich indenter. Each test consisted of 3 segments:

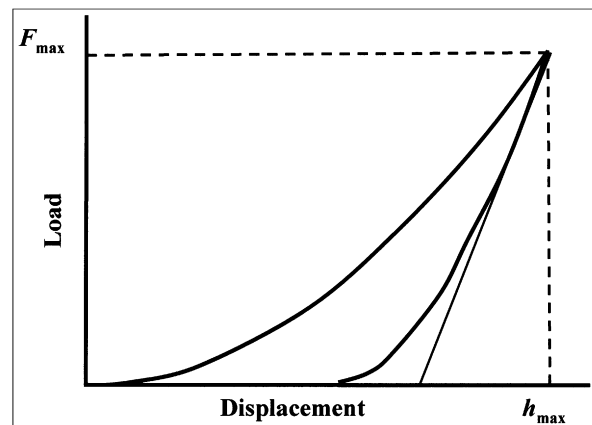


Fig 1. Schematic representation of the loading-unloading curve obtained by the nanoindentation testing. Linear extrapolation methods (International Organization for Standardization [ISO standard] 14577¹⁹) were used for unloading curves between 95% and 70% of F_{max} to calculate elastic modulus. F_{max} , maximum test force; h_{max} , maximum indentation depth.

10 seconds for loading to the peak value, 1 second holding at the peak load, and 10 seconds for unloading. Four peak loads of 2, 20, 50, and 100 mN were used for the measurements on the external surfaces, and only the 100-mN peak load was used for measurement on the cross-section surface. The hardness (H_{IT}) and elastic modulus (E_{IT}) were calculated by software available with the nanoindentation apparatus. Figure 1 shows a schematic representation of the loading-unloading curve obtained by the nanoindentation testing. Linear extrapolation methods (ISO standard 14577) were used for the unloading curve between 95% and 70% of the maximum test force to calculate the elastic modulus (Fig 1), and this method assumes that the first portion of the unloading curve is linear and extrapolates that linear portion to intersect the displacement axis.¹⁹ Load-frame compliance and tip-shape calibration were performed by using the method of Sawa and Tanaka²⁰ with fused quartz.

Vickers hardness measurements were performed at 25°C with a 2.94 N (300 gf) load and a 30-second dwell time for all specimens (MVK-F; Akashi, Tokyo, Japan). Ten indentations were made for each as-received wire product. The resulting values of Vickers hardness were then converted for comparison with the corresponding values from the nanoindentation test by using the following formula given in the ISO standard¹⁹: HV (Vickers hardness) = 0.0924 H_{IT} (hardness obtained by the nanoindentation test).

A 3-point bending test was carried out for the as-received wire products ($n = 10$). The span length

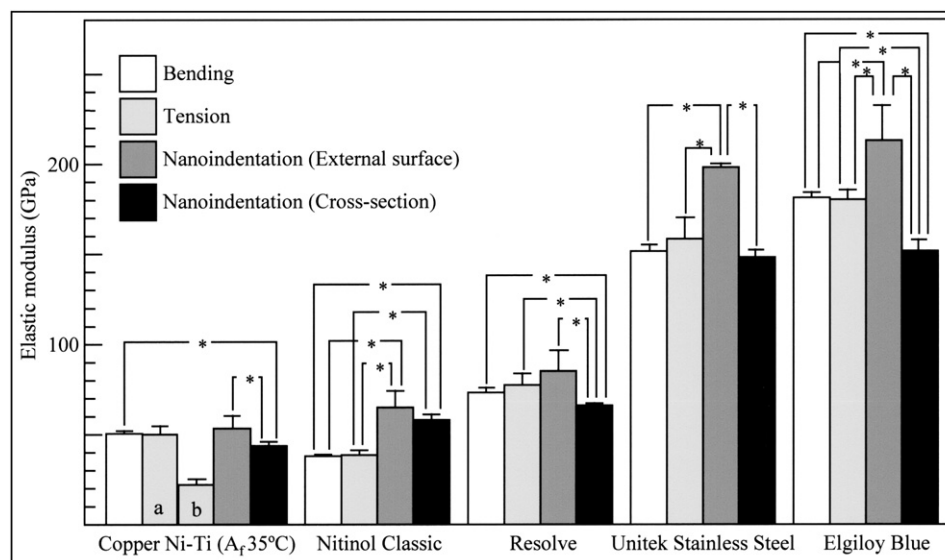


Fig 2. Comparison of elastic modulus (GPa) obtained in 3-point bending, tension, and nanoindentation tests. All archwires had dimensions of 0.016×0.022 -in. Two slopes were observed in the tension test plots for the 35°C Copper Ni-Ti; the first slope (a) was for austenitic nickel-titanium, and second slope (b) was for martensitic nickel-titanium. * $P < 0.05$ by the Mann-Whitney U test. The Copper Ni-Ti wire was excluded in the statistical comparisons of elastic modulus because the elastic deformation region of the tension test contained linear regions with 2 different slopes corresponding to the austenitic and martensitic phases.

between supports for the test specimens was 12 mm, which was chosen in accordance with ANSI/ADA specification no. 32.²¹ Specimens were loaded at 25°C to a deflection of 3 mm, by using a rate of 0.5 mm per minute with a universal testing machine (EZ Test; Shimadzu, Kyoto, Japan).

Tension tests for the as-received wire products were conducted with another universal testing machine (AG-X, Shimadzu) by using a 1-kN load cell ($n = 5$). A pair of grips with grooves was used for holding the wire specimens of approximately 30-mm gauge length, and the tension tests were carried out at a crosshead speed of 1 mm per minute. A video noncontact extensometer (DVE-201, Shimadzu) was used to measure the tensile elongation of the wires.

Statistical analysis

The data obtained from the 3-point bending and tension tests were analyzed with Trapezium 2 software (Shimadzu) to calculate the elastic modulus and 0.1% yield strength. Statistical comparisons were performed with the Statistical Package for Social Sciences software for Windows (version 16.0J; SPSS, Chicago, Ill). Because the data were not normally distributed, values of elastic modulus obtained with the 4 methods (3-point bending, tension, nanoindentation on external surface, and

nanoindentation on cross section of wire) were compared by using the Kruskal-Wallis test. The Mann-Whitney U test was then used for 2 independent groups, and the Bonferroni adjustment was applied. Values of yield strength obtained by the 3-point bending and tension tests were compared with the Mann-Whitney U test. Similarly, values of Vickers hardness and values of hardness obtained with the nanoindentation test for the external surfaces and the cross sections with the 100-mN load were compared by using the Kruskal-Wallis and Mann-Whitney U tests.

RESULTS

Mean values and standard deviations for the elastic modulus obtained with the 3-point bending, tension, and nanoindentation tests on the external and cross-section surfaces (100-mN peak load) are shown in Figure 2. The 35°C Copper Ni-Ti wire (Ormco, Orange, Calif), with an austenite-finish temperature A_r of 35°C, was excluded in the statistical comparisons of elastic modulus because the elastic deformation region of the tension test contained linear regions with 2 different slopes corresponding to the austenitic and martensitic phases. From the Kruskal-Wallis and Mann-Whitney U tests, the mean values of elastic modulus obtained by nanoindentation of the external surface for the Nitinol

Classic, Unitek Stainless Steel (both 3M Unitek, Monrovia, Calif), and Elgiloy Blue wires were significantly higher than those obtained by the conventional 3-point bending and tension tests. With the nanoindentation test and for all types of the wires except Nitinol Classic, the mean elastic modulus for the external surface was significantly higher than for the cross-section surface. The mean elastic modulus values obtained by nanoindentation of the cross-section surface for the Resolve (Dentsply GAC International, Bohemia, NY) and Elgiloy Blue wires were significantly lower than the mean values obtained with the bending and tension tests, although the mean elastic modulus obtained from nanoindentation of the cross-section surface for the Nitinol Classic was significantly higher than values obtained from the bending and tension tests. Although the different testing methods did not show identical results for elastic modulus, the order of the 5 products was the same.

Mean values and standard deviations of yield strength obtained from the conventional 3-point bending and tension tests are shown in Figure 3. For all types of wires (other than the martensitic phase of 35°C Copper Ni-Ti), the bending test showed significantly higher values than the tension test.

Mean values and standard deviations for mechanical properties (hardness and elastic modulus) obtained by the nanoindentation test on the external surfaces at 25°C are listed in Table II. Other than for the 35°C Copper Ni-Ti, the trend was for the mean hardness and elastic modulus for each wire to increase with decreasing indentation depth.

Mean values and standard deviations for Vickers hardness and the hardness of the external and cross-section surfaces obtained by the nanoindentation test with the 100-mN peak load are shown in Figure 4. Both mean values of hardness obtained with the nanoindenter (external and cross-section surfaces) for the Unitek Stainless Steel and Elgiloy Blue were significantly higher than the mean values for Vickers hardness, although both mean values of hardness for the 35°C Copper Ni-Ti were significantly lower than the mean value than for Vickers hardness. In nanoindentation testing, the mean values of hardness obtained from the cross-section surfaces were significantly higher than those from the external surfaces for Nitinol Classic, Resolve, and Unitek Stainless Steel.

DISCUSSION

Since hardness has relevance for mechanical properties such as yield strength and ultimate tensile strength, estimating the hardness of orthodontic wires is important, and microhardness testing has been used to

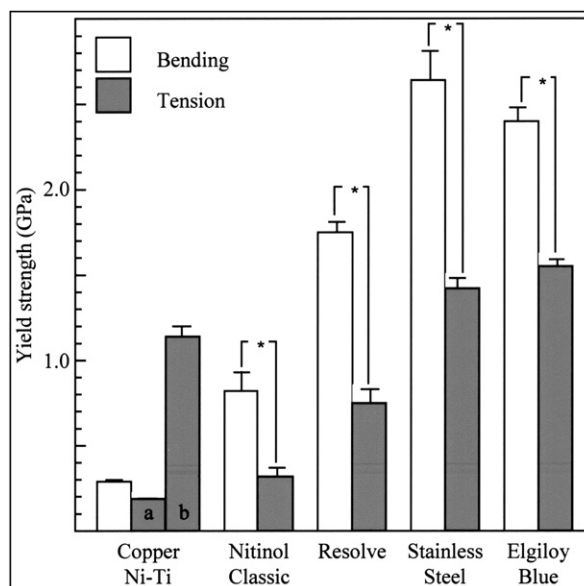


Fig 3. Comparison of yield strength (GPa) obtained in 3-point bending and tension tests. All archwires had dimensions of 0.016 × 0.022-in. Values for the 35°C Copper Ni-Ti correspond to the austenitic (a) and the martensitic (b) phases. * $P < 0.05$ by the Mann-Whitney U test. The Copper Ni-Ti wire was excluded in the statistical comparisons of yield strength because the elastic deformation region of the tension test contained linear regions with 2 different slopes corresponding to the austenitic and martensitic phases.

characterize orthodontic wires.^{1,9} A previous study also reported that the surface hardness of orthodontic wire affects frictional properties between the bracket and the archwire; this has major importance in clinical orthodontics.²² Mean values of Vickers hardness obtained in this study (391 kgf/mm² for Copper Ni-Ti, 408 kgf/mm² for Classic Nitinol, and 331 kgf/mm² for Resolve) were similar to those reported in a textbook on orthodontic materials (316 kgf/mm² for Copper Ni-Ti 428 kgf/mm² for Classic Nitinol, and 355 kgf/mm² for TMA [Ormco], another beta-titanium alloy).¹ Zinelis et al⁹ reported that the Vickers hardness of as-received Copper Ni-Ti was 367 kgf/mm², which was similar to our result. Alcock et al¹⁸ recently reported the effect of decontamination and clinical exposure on the elastic modulus and hardness of nickel-titanium and stainless steel wires (both from Dentsply GAC International) using the nanoindentation test. Their mean hardness values of 2.4 GPa for nickel-titanium wire and 4.1 GPa for stainless steel wire were lower than the values obtained in this study (3.3 GPa for Copper Ni-Ti and 6.1 GPa for Unitek Stainless Steel); this might be due to the different products used for each wire type in the 2 studies.

Table II. Summary of mechanical properties for orthodontic wires obtained by the nanoindentation test on external surfaces

Product	F_{max} (mN)	Hardness		Elastic modulus	
		Mean	SD	Mean	SD
		(GPa)		(GPa)	
Copper Ni-Ti (A _r 35°C)	100	3.3	0.8	53.4	7.2
	50	3.0	0.7	55.1	10.4
	20	3.3	0.8	56.5	11.0
	2	3.5	0.7	58.0	10.6
Nitinol Classic	100	4.9	1.2	65.0	9.4
	50	5.9	1.5	73.0	12.4
	20	6.4	2.1	77.0	14.1
	2	6.8	1.8	78.5	11.3
Resolve	100	3.1	0.7	85.1	11.1
	50	3.4	0.7	87.9	13.3
	20	3.7	1.0	91.0	14.5
	2	5.9	2.0	127.1	49.2
Unitek Stainless Steel	100	6.1	0.2	197.1	3.7
	50	6.2	0.6	202.7	11.3
	20	6.4	0.8	212.3	20.8
	2	8.4	1.5	257.6	38.6
Elgiloy Blue	100	6.9	0.9	213.0	19.9
	50	7.4	1.0	232.9	21.7
	20	7.8	1.7	226.1	33.4
	2	9.1	1.2	241.5	29.4

All values were obtained at 25°C. F_{max} , peak test load.

Mean values of elastic modulus obtained in this study were similar to those previously reported in the textbook on orthodontic materials (62–69 GPa for beta-titanium [TMA] wire, 160–180 GPa for stainless steel wire products from 2 companies, and 160–190 GPa for Elgiloy Blue wire).¹ Walker et al²³ reported a value of elastic modulus (132 GPa) obtained by 3-point bending for stainless steel wire (3M Unitek); this was lower than the value found in this study (151 GPa). On the other hand, the mean elastic modulus for stainless steel wire (3M Unitek) obtained by the nanoindentation test in this study was 197 GPa, which was lower (229 GPa) than that obtained from the nanoindentation test in a previous study for a different stainless steel wire product (Dentsply GAC International).¹⁸ The elastic modulus of a different nickel-titanium wire product (Dentsply GAC International) reported in this previous study was 71.5 GPa, which was higher than that found in our study (53 GPa) for 35°C Copper Ni-Ti.¹⁸ The differences in values for the same mechanical properties of each wire type are attributed to the different products evaluated and the different temperatures for the nanoindentation tests (25°C in this study and 28°–32°C in the study of Alcock et al¹⁸).

Previous studies with conventional differential scanning calorimetry²⁴ and temperature-modulated differential scanning calorimetry²⁵ showed that the nonsuperelastic alloy Nitinol Classic is entirely or almost entirely martensite at room temperature, whereas the superelastic 35°C Copper Ni-Ti is almost entirely austenite at room temperature in addition to a small amount of R-phase. In our study, both measurements of hardness (Vickers hardness and hardness obtained by the nanoindentation testing) for the Nitinol Classic alloy showed higher values than for the 35°C Copper Ni-Ti; this can be explained by the heavily cold-worked martensite phase in Nitinol Classic.¹ The 2 slopes observed with the tension test for the 35°C Copper Ni-Ti were associated with the austenitic phase at lower stress and the martensitic phase at higher stress. It was previously found that the martensite phase in nickel-titanium orthodontic wires has a lower elastic modulus than the austenite phase.² In this study, the elastic modulus of Nitinol Classic was intermediate between values for the martensite and austenite phases of the 35°C Copper Ni-Ti; this might be explained by the presence of some austenite in Nitinol Classic or by different levels of work hardening for the martensite in the 2 wire alloys.

For all types of wires, the mean values of yield strength obtained in 3-point bending were significantly higher than those obtained with the tension test; this trend agrees with a previous study.³ The reason for this behavior is the differing natures of the 2 modes of deformation.³ The outermost portion of the specimen's cross section initially undergoes permanent deformation during bending while the rest of the specimen is still within the elastic range, whereas plastic deformation over the entire cross-section begins at the same stress during the tension test.

In this study, the hardness obtained by the nanoindentation test was found to increase with decreasing indentation depth. (The maximum indentation depth for the 100-mN peak load varied for the 4 wire types, but did not exceed 1500 nm.) Since the orthodontic wire alloys form native oxide films, yielding under the nanoindenter might be governed by fracture of the oxide film rather than onset of plastic deformation in the metal itself, and the oxide thickness thus affects the mechanical properties.¹³ On the other hand, it has been reported that the hardness of pure silver increases with decreasing indentation size to depths as small as 30 nm, and that differences in dislocation density in the regions surrounding an indentation have an effect on the hardness, but only within a specific range of indentation depths (about 50–250 nm).¹¹ Consequently, a possible reason that the hardness obtained by the nanoindentation test in this study increased with decreasing indentation depth is that the hardness of the oxide film formed on

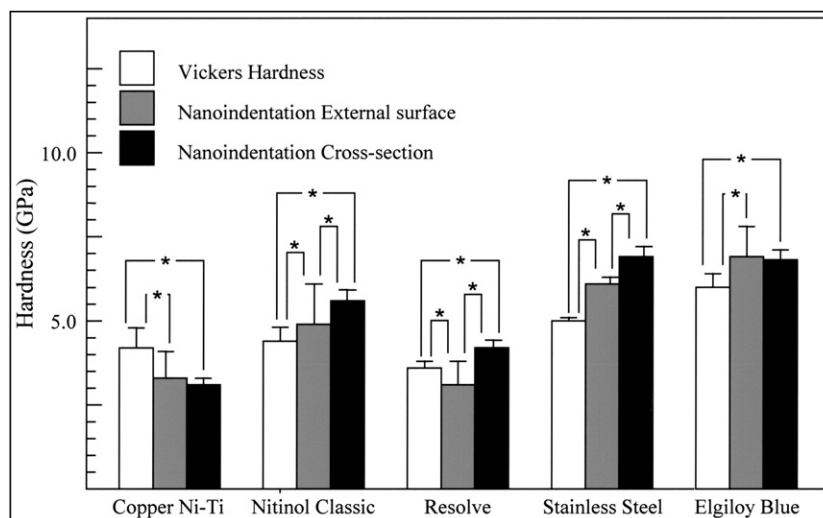


Fig 4. Vickers hardness and hardness obtained by the nanoindentation test for the external and cross-section surfaces. All values were obtained at 25°C. Measured values of Vickers hardness were converted for comparison to hardness obtained by the nanoindentation test by using the formula, $HV = 0.0924 H_{IT}$, available in the ISO standard.¹⁹ * $P < 0.05$ by the Mann-Whitney U test.

the wire surface was greater than that for the bulk alloy. However, for our nanoindentation testing, the mean hardness values obtained from the cross-section surfaces were significantly higher than those obtained from the external surfaces for Nitinol Classic, Resolve, and Uitek Stainless Steel. Since a native oxide layer should also form rapidly on the cross-section surfaces, these differences in hardness for the external and cross-section surfaces might instead be due to differences in work hardening that occur over the cross section during wire processing.¹ The rectangular wires used in this study were created by rolling initially round wires that had previously been subjected to a series of drawing and intermediate heat-treatment steps. In addition, the mechanical polishing step for preparation of the cross-section specimens can alter the work hardening in the original wire specimen.¹⁹

The elastic modulus of stainless steel and nickel-titanium orthodontic wires can be affected by the level of cold working. For both wire alloys, this mechanical deformation causes a martensite phase with lower values of elastic modulus to form from the austenite phase having a higher elastic modulus.^{1,2,26} The martensite phase in nickel-titanium wire alloys can also experience substantial work hardening, which was found for Nitinol Classic wires.¹ Further research is needed to understand the potentially complex relationships between microstructural variations and mechanical properties obtained at the surfaces and over the cross sections of orthodontic wires.

The main reason for the differences in mechanical properties obtained with the nanoindenter and the

conventional mechanical property tests (Vickers hardness, bending, and tension tests) for orthodontic wires is that the nanoindentation test samples a much smaller volume of material than does the Vickers microhardness test. Moreover, the stress distribution at the nanoindenter is complex compared with the much simpler stress distributions for the macroscopic tension and bending tests.

The nanoindentation test is convenient for orthodontic alloys, because it requires only a small volume of specimen material, and it is possible to obtain both hardness and elastic modulus at the same time. This test is also a suitable method for the evaluation of near-surface mechanical properties for other important materials in dentistry, such as enamel, dentin, composite resins, cements, ceramics, and case alloys. There might be notable differences between the near-surface mechanical properties and bulk mechanical properties that have important clinical relevance.

CONCLUSIONS

Under the conditions of this study, the following conclusions can be drawn.

1. Three testing methods (bending, tension, and nanoindentation tests) did not yield identical values of elastic modulus and hardness, although the order among the 5 wire products was always the same.
2. Variations in the results for the nanoindentation and conventional mechanical property tests can be attributed to the different material volumes

- sampled, the different work-hardening levels, and perhaps the effect of the oxide layer on the surface.
3. The mean values of yield strength obtained in 3-point bending were significantly higher than those obtained with the tension test.
 4. Mean values of hardness and elastic modulus obtained by the nanoindentation test for the wires were found to generally increase with decreasing indentation depth.
 5. Mean hardness values obtained from the cross-section surface were significantly higher than those obtained from the external surface for Nitinol Classic, Resolve, and Unitek Stainless Steel.

We thank Junzo Suzuki and Takashi Murakami at Shimadzu for kindly performing the tension tests.

REFERENCES

1. Brantley WA. Orthodontic wires. In: Brantley WA, Eliades T, editors. Orthodontic materials: scientific and clinical aspects. Stuttgart, Germany: Thieme; 2001. p. 77-104.
2. Kusy RP. A review of contemporary archwires: their properties and characteristics. *Angle Orthod* 1997;67:197-208.
3. Asgharnia MK, Brantley WA. Comparison of bending and tension tests for orthodontic wires. *Am J Orthod* 1986;89:228-36.
4. Miura F, Mogi M, Ohura Y, Hamanaka H. The super-elastic property of the Japanese NiTi alloy wire for use in orthodontics. *Am J Orthod Dentofacial Orthop* 1986;90:1-10.
5. Khier SE, Brantley WA, Fournelle RA. Bending properties of superelastic and nonsuperelastic nickel-titanium orthodontic wires. *Am J Orthod Dentofacial Orthop* 1991;99:310-8.
6. Meling TR, Odegaard J. The effect of short-term temperature changes on superelastic nickel-titanium archwires activated in orthodontic bending. *Am J Orthod Dentofacial Orthop* 2001;119:263-73.
7. Iijima M, Ohno H, Kawashima I, Endo K, Mizoguchi I. Mechanical behavior at different temperatures and stresses for superelastic nickel-titanium orthodontic wires having different transformation temperatures. *Dent Mater* 2002;18:88-93.
8. Kohn DH. Mechanical properties. In: Craig RG, Powers JM, editors. Restorative dental materials. 11th ed. St Louis: Mosby; 2002. p. 67-124.
9. Zinelis S, Eliades T, Pandis N, Eliades G, Bourauel C. Why do nickel-titanium archwires fracture intraorally? Fractographic analysis and failure mechanism of in-vivo fractured wires. *Am J Orthod Dentofacial Orthop* 2007;132:84-9.
10. Dieter GE. Mechanical metallurgy. 3rd ed. New York: McGraw-Hill; 1986. p. 329-32.
11. Pharr GM, Oliver WC. Nanoindentation of silver-relations between hardness and dislocation structure. *J Mater Res* 1989;4:94-101.
12. Oliver WC, Pharr GM. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J Mater Res* 1992;7:1564-83.
13. Bahr DF, Kramer DE, Gerberich WW. Non-linear deformation mechanisms during nanoindentation. *Acta Mater* 1998;46:3605-17.
14. Fischer-Cripps AC. Contact mechanics: factors affecting nanoindentation test data. In: Fischer-Cripps AC, editor. Nanoindentation (Mechanical Engineering Series). New York: Springer; 2002. p. 1-19: 61-82.
15. Dickinson ME, Wolf KV, Mann AB. Nanomechanical and chemical characterization of incipient in vitro carious lesions in human dental enamel. *Arch Oral Biol* 2007;52:753-60.
16. He LH, Swain MV. Influence of environment on the mechanical behaviour of mature human enamel. *Biomaterials* 2007;28:4512-20.
17. Iijima M, Muguruma T, Brantley WA, Yuasa T, Uechi J, Mizoguchi I. Effect of mechanical properties of fillers on the grindability of composite resin adhesives. *Am J Orthod Dentofacial Orthop* 2010;138:420-6.
18. Alcock JP, Barbour ME, Sandy JR, Ireland AJ. Nanoindentation of orthodontic archwires: the effect of decontamination and clinical use on hardness, elastic modulus and surface roughness. *Dent Mater* 2009;25:1039-43.
19. International Organization for Standardization. ISO 14577-1. Metallic materials—instrumented indentation test for hardness and materials parameters, part 1: test method. 1st ed. Geneva: International Organization for Standardization; 2002.
20. Sawa T, Tanaka K. Simplified method for analyzing nanoindentation data and evaluating performance of nanoindentation instruments. *J Mater Res* 2001;16:3084-96.
21. American National Standard/American Dental Association (ANSI/ADA). Specification no. 32. Orthodontic wires 2000. Available at: <http://www.ada.org/830.aspx>. Accessed May 10, 2011.
22. Saunders CR, Kusy RP. Surface topography and frictional characteristics of ceramic brackets. *Am J Orthod Dentofacial Orthop* 1994;106:76-87.
23. Walker MP, Ries D, Kula K, Ellis M, Fricke B. Mechanical properties and surface characterization of beta titanium and stainless steel orthodontic wire following topical fluoride treatment. *Angle Orthod* 2007;77:342-8.
24. Bradley TG, Brantley WA, Culbertson BM. Differential scanning calorimetry (DSC) analyses of superelastic and nonsuperelastic nickel-titanium orthodontic wires. *Am J Orthod Dentofacial Orthop* 1996;109:589-97.
25. Brantley WA, Iijima M, Grentzer TH. Temperature-modulated DSC provides new insight about nickel-titanium wire transformations. *Am J Orthod Dentofacial Orthop* 2003;124:387-94.
26. Khier SE, Brantley WA, Fournelle RA. Structure and mechanical properties of as-received and heat-treated stainless steel orthodontic wires. *Am J Orthod Dentofacial Orthop* 1988;93:206-12.