Low temperature polytetrafluoroethylene (PTFE) coating improves the appearance of orthodontic wires without changing their mechanical properties

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A strong esthetic demand exists for white-colored rather than metallic-colored dental appliances. Polytetrafluoroethylene (PTFE), which is chemically stable with extremely low friction, is a suitable white-colored coating for dental appliances. In the conventional PTFE coating process, base materials are heated to approximately 400°C, which could change their mechanical properties. Examination of the PTFE-coating of stainless steel, nickel titanium (Ni-Ti), and β-titanium (Ti-Mo and Ti-Nb) revealed that the conventional 380°C coating, but not the newly developed low temperature coating at 200°C, reduced elasticity and bendability, and changed the crystal structure, especially in Ni-Ti and Ti-Nb wires. PTFE-coating at 200°C resulted in less discoloration, microbial adhesion, and friction against brackets, and higher tolerance of wear than did the conventional 380°C coating. These results suggest that low temperature PTFE coating is an excellent method for improving the metallic appearance of orthodontic wires without changing their mechanical properties.

Keywords: Mechanical strength, X-ray diffraction analysis, Friction, Wear test, Bacteria adhesion

INTRODUCTION

In recent times, orthodontic patients in Japan have tended to prefer clear or white-colored brackets made of ceramic or plastic resin1 to metallic-colored appliances. Esthetic orthodontic wires are usually fabricated by coating the surface of metallic wires with white-colored resins, such as epoxy resin, polyurethane, and polytetrafluoroethylene (PTFE). PTFE is chemically stable, non-toxic to humans, highly resistant to heat and chemicals, and has an extremely low coefficient of friction2,3. Therefore, PTFE is widely used in medical applications, and is especially suitable for orthodontic appliances including wires. We previously investigated the application of PTFE molding in prostheses for provisional and/or permanent use, and in dental instruments/devices. We established that PTFE provides excellent impact absorption, high wear resistance for maintenance of occlusal vertical dimension and original function, and ease of cleaning, which could lead to better oral hygiene and health, improved oral frailty, better quality of life, and an extended healthy life expectancy4. In the process of PTFE coating rather than molding, the high temperature exposure during coating might alter the mechanical characteristics of their underlying base metallic materials5.

During orthodontic treatment, the large difference in elasticity between nickel titanium (Ni-Ti) and stainless steel (SUS) wires causes some clinical problems, such as brackets detaching when the wire is changed from Ni-Ti wires to SUS wires, resulting in pain for the patient. In such cases, β-titanium wires are more suitable because they are bendable and exhibit the mechanical property of high elasticity6. Titanium wires (both Ni-Ti and β-titanium wires) are corroded by fluoride7-10. Approximately half the content of Ni-Ti wire is nickel, which tends to cause allergic and toxic reactions, whereas β-titanium wire do not contain nickel. However, β-titanium wire is expensive, and deteriorates rapidly under physical stress, such as heating and repeated stress11,12.

This study investigated the physical properties of orthodontic wires composed of SUS, Ni-Ti, and β-titanium (Ti-Mo13,14 and Ti-Nb) that were coated with PTFE at a low temperature (200°C) and at a conventional temperature (380°C). The physical properties examined were mechanical characteristics, crystal structure, discoloration with foods and drinks, microbial adhesion, friction against metallic brackets, and wear/detachment of the coating film with toothbrushing.

MATERIALS AND METHODS

Materials

We prepared SUS, Ni-Ti, and β-titanium (Ti-Mo13,14 and Ti-Nb) orthodontic wires for PTFE coating. SUS wires used in this research (Permachrome standard, 3M Oral...
Care, St. Paul, MN, USA) were made of SUS304. Round wires with cross-sectional size of 0.018″ were used for the three-point bending test, and rectangular wires with cross-sectional size of 0.017″x0.025″ were used for the other tests. Ni-Ti wires (Sentalloy™ upper standard, TOMY International, Tokyo, Japan), Ti-Mo wires (BENDALOY™, Rocky Mountain Morita [currently known as JM Ortho], Tokyo, Japan), and Ti-Nb wires (GUMMETAL™, JM Ortho, Tokyo, Japan) were used in this study.

For the surface wear test, coloration test, and microbial adhesion test, SUS plates of SUS304 measuring 2 cm×2 cm×2 mm were prepared. Composite resin plates (CR; Ceramage, Shofu, Kyoto, Japan) measuring 2 cm×2 cm×2 mm were also prepared according to the manufacturer’s instructions as a positive control for the coloration test.

These materials with or without PTFE coating were prepared for each experiment (Fig. 1).
**PTFE coating**

Specimens of each material were coated with PTFE at low temperature (200°C) or conventional temperature (380°C) at a specialized factory (Fluoro Coat, Kawagoe, Japan). Conventional PTFE-coating consisted of two spraying stages (Fig. 2). The wires were cleaned and degreased by wiping with paper immersed in isopropyl alcohol. The labial/buccal side of the degreased wires was blasted with #200 abrasive sprayed through a suction-type blast machine (B-2, Akiyama Sangyo, Osaka, Japan) for 5 s, and primer (L-4062GB, Fluoro Coat) was sprayed onto the wire surface with a spraying pressure of 0.2 MPa for 30 min to increase adhesion. The wires were then dried again at 100°C for 10 min. The coated wires were baked at 380°C for 20 min in a drying furnace for fixation, and air-cooled at room temperature for 10 min.

**Measurement of film-thickness and surface roughness**

The PTFE film thickness was measured using a micrometer (GM001, Mitsutoyo, Kawasaki, Japan), i.e. difference of wire width between before and after PTFE coating (Fig. 1). The surface roughness, consisting of Ra (arithmetic average roughness) and Rz (maximum height roughness), was measured at the center of the surface of each specimen (measurement length: 3 mm, speed: 0.300 mm/s, cutoff wavelength: 0.800 mm) with a surface texture and contour integrated measuring instrument (Surfcom 590A, Tokyo Seimitsu, Tokyo, Japan) according to the JIS-'94 method (Fig. 1).

**Mechanical strength and right-angle bending test**

To confirm the flexural strength (bending strength) and elastic modulus of each orthodontic wire with or without heating of the PTFE coating, we performed a three-point bending test according to JIS T 6530: 2017 using a universal testing machine (Autograph AGS-J, Shimadzu, Kyoto, Japan) at crosshead speeds of 1.25 and 5.0 mm/min with strokes of 1.5 and 3.1 mm for bendable wires (SUS and β-titanium) and Ni-Ti wires, respectively (Fig. 1). The rated capacity of the load cell used in this study was 10 kN. The support span in this test was set at 10 mm. A stress-strain curve was recorded for each specimen, and flexural strength, elastic modulus, and permanent deformation were calculated and determined from each record. The bending stress \( \delta \) (MPa) for round wires was calculated by means of the following equation:

\[
\delta = \frac{8P}{\pi d^3}
\]

\( P \) is the flexural load, \( l \) is support support span, and \( d \) is diameter of orthodontic wires.

The elastic modulus (MPa) was calculated from the elastic range of the stress-strain plot according to the following formula:

\[
\text{Elastic modulus (MPa)} = \frac{\Delta \text{stress} (\delta)}{\Delta \text{strain}}
\]

A right-angle bending test was performed to clarify the number of bends possible until fracture of the rectangular wires with or without PTFE coating after 90°-repeated bending with square beaked orthodontic pliers (Light wire plier, Task, Tokyo, Japan) with one bending for 1 s with 1 s intervals (Fig. 1).

**X-ray diffraction (XRD) analysis**

Before XRD analysis, specimens with or without PTFE coating had the colored oxide film on their metallic surfaces removed by SiC-paper #400 and #800 to expose their metallic-colored surfaces. The specimens, i.e. bundled wires, were then washed under running water, followed by distilled water, acetone, and ethanol.

The crystal structural changes of the washed wire
specimens that had been heated during PTFE-coating were examined by XRD using an X-ray diffractometer (RINT-UltimaIII, Rigaku, Tokyo, Japan). XRD analysis was performed with Cu Kα radiation at 40 kV and 40 mA at 2θ/0-scanning mode, with the angle of the incident X-ray beam (θ) from 15° to 45° scanned speed at 1.0°/min with scanning step at 0.02°. To evaluate the deduced θ phase of Ti-Nb wires at 75–85°, we obtained the eight-times integrated data of eight-times measurement at 2θ/0-scanning mode with 0.02° step for 4s (Fig. 1).

Friction test between PTFE-coated wires and a bracket
Friction between wires and a bracket was applied with a Rheometer (NRM-2010J-CW, Fudoh Kogyo, Tokyo, Japan) using a jig fixed to a bracket at 0° inclined to the longitudinal direction of wires, which were set 3 cm from the wire-fixed jig15 (Fig. 1). The testing machine was operated six times at the same moving distance of 1 cm and a speed of 1 mm/s. The bracket applied to this experiment was a standard edgewise bracket (Standard Edgewise Mesh brackets, Tomy International) tied to each wire with an elastomeric module (ToughO, JM Ortho). Straight wires of SUS and β-titanium (not arch formed wires) of 0.017″×0.025″ sectional size and 10 cm length, were used for this experiment. Before the experiment, we searched for straight-formed Ni-Ti wires of 0.017″×0.025″ sectional size, but they were not available in Japan. We also considered using the straight premolar–molar portion of the arch formed wires; however, we could not find such wires with a straight portion longer than 10 cm. Moreover, Ni-Ti wires were flexible, and could be easily curved by the elastic modules tying them to a bracket. For these reasons, we used SUS and β-titanium straight wires, but not Ni-Ti wires, for this experiment.

Detachment test and wear test against toothbrushing
Before evaluating the detached and worn portions of the wires (the detachment and wear test)16, each specimen surface was brushed with a toothbrush (Dent. EX Slimhead II 33M [footprint of bristle: width 6.0 mm, length 22.4 mm, and bristle length: 9.5 mm], Lion, Tokyo, Japan) under 1.96 N (200 gf) for 27 min by a brushing machine, which was attached to a strain gauge (F-01 W-12 T11 W3, Minebea Mitsumi, Nagano, Japan) to monitor brushing pressure. The test was performed with a brushing distance of 10 mm at 4 Hz (240 rpm) with a 90° brushing angle, and the toothbrush moved the sectional direction of wires (90° of the longitudinal axis of the wires) (Fig. 1) because the toothbrush bristles missed the wire/coated surfaces when the toothbrush was moved to the longitudinal direction of the wires in a preliminary experiment. This test condition was decided according to the stored toothbrushing data at Nippon Dental University College at Niigata. Because 28 teeth with 56 tooth surfaces (labial/buccal and lingual sides) in each oral cavity were assumed to be brushed by the scrubbing method, 28 parts existed in each oral cavity from each toothbrushing stroke for two teeth. If one toothbrushing period takes 180 s (3 min), it takes approximately 6 s to brush each part, making a total of 18 s/day when brushing three times each day, 540 s (9 min)/month, and 27 min/3 months. After 6,480 cycles/27 min of brushing, the specimen surface was thoroughly cleaned with a water spray and then the abrasive slurry was reapplied to the surface. The toothbrush that had been used was replaced with a new toothbrush after testing of each specimen.

To estimate the amount of detachment of the PTFE coating after toothbrushing, each wire was observed under a stereoscopic microscope (SMZ800, Nikon, Tokyo, Japan) at ×30 magnification and the number of detached portions of the PTFE coating was counted (Fig. 1).

The PTFE-coated surfaces of the SUS plates and orthodontic wires before and after the wear test were observed with a scanning electron microscope (VE-8800, KEYENCE, Osaka, Japan) at an acceleration voltage of 2 kV according to the manufacturer’s instructions.

Coloration test
Before the coloration test, the surface of the SUS and CR plates was polished with waterproof sandpaper #1200 (C34P, Riken Corundum, Saitama, Japan). To measure color, each specimen (10×10×2 mm) was immersed in 10 mL of distilled water (DW; control), coffee solution (1 g of Nescafe Excella coffee powder in 7 mL of DW, Nestle Japan, Hyogo, Japan), tea solution (1 g of Lipton Yellow Label tea leaves in 75 mL of DW, Unilever Japan, Tokyo, Japan), and curry solution (1 g of S&B curry powder in 20 mL of DW, S&B Foods, Tokyo, Japan) for 48 h at 37°C17. The specimens were then washed twice with DW, and dried in the dark at room temperature overnight. The color of the intact and dried specimens was evaluated using a colorimeter (CR-100, Minolta, Tokyo, Japan) following calibration against the standard white porcelain plate provided (Fig. 1). We measured \( L^* \) (lightness of color; \( L^*=0 \) indicates black and \( L^+=100 \) indicates diffuse white), \( a^* \) (position between red/magenta and green; negative values indicate green and positive values indicate magenta), and \( b^* \) (position between yellow and blue; negative values indicate blue and positive values indicate yellow), and then calculated \( \Delta E^{*ab} \) (\( \Delta E \)) using CIELAB18,19. Color differences between the control (immersed in DW) and specimens colored with drink/food solution were exhibited as \( \Delta E \), which was calculated using the following equation:

\[
\Delta E^{*ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}
\]

The color difference (\( \Delta E \)) was defined by the NBS unit (National Bureau of Standards, National Institute of Standards and Technology, Gaithersburg, MD, USA) as follows: 0–0.5; trace, 0.5–1.5; slight, 1.5–3.0; noticeable, 3.0–6.0; appreciable, 6.0–12.0; high, 12.0–; very high. By comparing the \( \Delta E \) value of specimens colored with drink/food solution we were able to establish the extent of coloration of each specimen.
**Bacterial adhesion test**

*Streptococcus (S) mutans* and *S. sanguinis* were cultured in 1% glucose added to brain heart infusion medium (BHI; Beckton, Dickinson, Franklin Lakes, NJ, USA) at 37°C. Solutions of *S. mutans* and *S. sanguinis* (200 µL of OD 0.6 solution) were placed on each specimen, and cultured for 2 h at 37°C. Specimens were washed twice with PBS after cultivation, and reacted with Alamar Blue® solution (Thermo Fisher Scientific, Tokyo, Japan) for 2 h at 37°C. These reacted solutions were placed in 96-well plates, and the fluorescence was measured (excitation wavelength 540 nm; emission wavelength 590 nm) with a microplate reader (Powerscan MX, DS Pharma Biomedical, Osaka, Japan) to evaluate the reducing activity of living bacteria adhering to each specimen (Fig. 1).

**Experimental conditions, data and statistical analysis**

All experiments were performed in a laboratory environment maintained at 22±1°C. Each experiment was repeated eight times independently, with the maximum and minimum values in each data set being removed before calculation of mean values. Data are given as the mean±standard deviation. The statistical significance of the differences within and between groups was determined with the Tukey-Kramer test, comparing all columns. Statistical significance was accepted at *p*<0.01. All statistical analysis was performed with EZR (Saitama Medical Center, Jichi Medical University, Saitama, Japan), which is a graphical user interface for R (The R Foundation for Statistical Computing; Vienna University of Technology, Vienna, Austria) on a workstation computer (MB-P5300X-WS, Mouse, Tokyo, Japan).

**RESULTS**

First, we noted appearance changes in the PTFE-coated wires by visual observation (Fig. 3). Heating to 380°C during PTFE coating altered the metallic surfaces of β-titanium wires (Ti-Mo and Ti-Nb). In particular, the metallic surface color of Ti-Nb wires markedly darkened by heating to 380°C (Fig. 3). The PTFE-coated surfaces of orthodontic wires heated to 200°C and 380°C were observed with a scanning electron microscope (Fig. 4), and the film thickness and surface roughness of their coating was measured (Tables 1 and 2). Film thickness of PTFE coating at 380°C was thicker than that of 200°C coating, and was exhibited approximately 30–45 µm (Table 1). Scanning electron microscopic observation of the surface of the 200°C coated wires revealed greater unevenness of the particles compared with those of the 380°C group, which was consistent with the surface roughness measurements (Fig. 4 and Table 2).

Changes in the mechanical characteristics of wires caused by heating were examined by the three-point bending test and the right-angle bending test (Fig. 1). The three-point bending test showed that SUS, Ni-Ti, and β-titanium coated wires (especially Ti-Nb wires) heated to 380°C had a higher elastic modulus and maximum stress than those heated to 200°C (Fig. 5 and Table 3). These results were supported by the right-angle bending test that indicated the number of bends that could be completed until wire fracture (Fig. 6 and Table 4). In Ni-Ti wires, 380°C heating resulted in a loss of superelasticity with permanent deformation (Fig. 5). In Ti-Mo wires, 200°C heating did not alter their mechanical properties, whereas 380°C heating resulted in slight changes. Ti-Nb wires with PTFE coating at 200°C were bendable to the same level as non-coated Ti-Nb wires, but Ti-Nb wires with PTFE coating at 380°C

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**Fig. 3** PTFE-coated orthodontic wires.

The PTFE-coated side (labial/buccal side of wires) is situated on the lower side of the wires in the photos. Non-coated wires are intact products without coating or heating. The white line represents 500 µm.
Table 1  Film-thickness of PTFE-coating on rectangular wires

| Material   | PTFE-coating temperature (°C) | Film thickness (µm) |
|------------|-------------------------------|---------------------|
|            | 200                           | 30.33± (7.916)      |
|            | 380                           | 46.83± (3.601)      |
| SUS        | 200                           | 30.30± (7.890)      |
|            | 380                           | 28.51± (5.822)      |
| Ni-Ti      | 200                           | 28.50± (11.059)     |
|            | 380                           | 43.06± (10.621)     |
| β-titanium | 200                           | 27.33± (9.689)      |
|            | 380                           | 44.52± (3.209)      |

n=6 for each experiment. Values are mean and (S.D.).
Ti-Mo; Ti-11.5Mo-6Zr-4.5Sn, Ti-Nb; Ti-23Nb-0.7Ta-2.0Zr-1.2O
Superscript letters denote statistically significant differences (p<0.01).

Table 2  Surface roughness of PTFE-coated rectangular wires

| Material   | PTFE coating (°C) | Surface roughness (µm) |
|------------|-------------------|------------------------|
|            |                   | Ra     | Rz     |
| SUS        | Non              | 0.02±  (0.008) | 0.11± (0.013) |
|            | 200              | 1.51±  (0.350) | 11.26± (2.151) |
|            | 380              | 0.53±  (0.045) | 4.24± (0.873)  |
| Ni-Ti      | Non              | 0.21±  (0.025) | 1.34± (0.167)  |
|            | 200              | 1.27±  (0.088) | 9.26± (1.247)  |
|            | 380              | 0.54±  (0.059) | 5.44± (0.618)  |
| β-titanium | Ti-Mo            | 0.18±  (0.012) | 1.13± (0.218)  |
|            | 200              | 1.36±  (0.281) | 8.23± (1.769)  |
|            | 380              | 0.58±  (0.045) | 5.30± (0.282)  |
|            | Ti-Nb            | 0.17±  (0.015) | 1.08± (0.196)  |
|            | 200              | 1.09±  (0.092) | 8.37± (1.646)  |
|            | 380              | 0.53±  (0.062) | 6.53± (1.228)  |

n=6 for each experiment. Values are mean and (S.D.).
Ti-Mo; Ti-11.5Mo-6Zr-4.5Sn, Ti-Nb; Ti-23Nb-0.7Ta-2.0Zr-1.2O, Non; intact products without PTFE coating or heating
Superscript letters denote statistically significant differences (p<0.01) within each material of each measurement item.

could not be bent once at 90° because of their fracturing (Table 4).
The crystal structural changes in the metallic surfaces of specimens heated during PTFE coating were examined by XRD, which confirmed the results of the mechanical property changes (Figs. 5, 7 and Tables 3, 4). XRD profiles of SUS wires heated at 200°C and 380°C were the same as those of non-heated SUS wires without PTFE-coating. In contrast to intact SUS powder, each SUS wire exhibited a deformation-induced martensitic phase as a body-centered cubic structure diffraction peak at a 45° diffraction angle as well as an austenite phase as a parent phase at a 43° diffraction angle (23) (Fig. 7A). In Ni-Ti wires, specimens heated to 380°C, which diminished superelasticity, exhibited a martensitic phase instead of an austenite phase at a 42° diffraction angle in unheated and 200°C heated specimens (24,25) (Fig. 7B).
Fig. 5 Stress–strain curves of PTFE-coated wires.
A. SUS wires, B. Ni-Ti wires, C. β-titanium wires 1) Ti-Mo-Zr-Sn and 2) Ti-Nb-Ta-Za-O. Non-coated wires are intact products without coating or heating. Solid lines denote non-coated wires, dotted lines denote 200°C PTFE-coated wires, and wide-dotted lines denote 380°C PTFE-coated wires.

Table 3 Mechanical characteristics of PTFE-coated wires

| Material       | PTFE coating (°C) | Sectional size (mm) | Stroke (mm) | Elastic modulus (MPa) | Maximum stress (MPa) | Permanent deformation (mm) |
|----------------|-------------------|---------------------|-------------|-----------------------|----------------------|-----------------------------|
| SUS            | Non               | 0.456ϕ (0.018″)     | 1.5         | 4,559.5a (36.02)      | 4,472.9a (19.58)      | 0.560a (0.0025)             |
|                | 200               | 0.456ϕ (0.018″)     | 1.5         | 4,592.8a (51.86)      | 4,435.3a (35.03)      | 0.562a (0.0361)             |
|                | 380               | 0.456ϕ (0.018″)     | 1.5         | 4,865.6a (67.29)      | 4,616.3a (30.61)      | 0.518a (0.0053)             |
| Ni-Ti          | Non               | 0.443ϕ (0.018″)     | 3.1         | 995.9a (10.74)        | 833.3a (30.77)        | 0.001a (0.0002)             |
|                | 200               | 0.443ϕ (0.018″)     | 3.1         | 992.6a (23.92)        | 891.7a (35.84)        | 0.003b (0.0008)             |
|                | 380               | 0.443ϕ (0.018″)     | 3.1         | 1,027.8a (15.94)      | 744.8a (26.56)        | 1.589a (0.0350)             |
| Ti-Mo          | Non               | 0.439ϕ (0.018″)     | 1.5         | 2,174.2a (15.83)      | 2,231.1a (28.28)      | 0.419a (0.0016)             |
|                | 200               | 0.439ϕ (0.018″)     | 1.5         | 2,179.0a (20.11)      | 2,261.6a (14.62)      | 0.425b (0.0059)             |
|                | 380               | 0.439ϕ (0.018″)     | 1.5         | 2,271.4a (23.25)      | 2,383.4a (19.59)      | 0.406b (0.0052)             |
| Ti-Nb          | Non               | 0.451ϕ (0.018″)     | 1.5         | 1,663.4a (11.01)      | 1,991.6a (16.00)      | 0.156a (0.0036)             |
|                | 200               | 0.451ϕ (0.018″)     | 1.5         | 1,705.3b (14.12)      | 2,034.2b (18.22)      | 0.169b (0.0050)             |
|                | 380               | 0.451ϕ (0.018″)     | 1.5         | 1,844.5c (18.13)      | 2,250.8c (14.14)      | 0.192c (0.0037)             |

n=6 for each experiment. Values are mean and (S.D.).

Ti-Mo; Ti-11.5Mo-6Zr-4.5Sn, Ti-Nb; Ti-23Nb-0.7Ta-2.0Zr-1.2O, Non; intact products without PTFE coating or heating. Superscript letters denote statistically significant differences (p<0.01) within each material of each measurement item.

Similar to Ni-Ti wires, the XRD profiles of unheated and 200°C heated Ti-Mo wires were almost identical. The XRD profiles of 380°C heated Ti-Mo wire exhibited almost the same pattern as unheated and 200°C heated Ti-Mo wires; however, each diffraction peak of the 380°C heated wire was markedly higher, especially the peak at a 56° diffraction angle (Fig. 7 C-1). The XRD profiles of unheated and 200°C heated Ti-Nb wires were almost identical. The XRD profiles of the 380°C heated Ti-Nb wire followed almost the same pattern as unheated and
Fig. 6 Right-angle bending test.

A. Bending process of non-coated SUS wire. The condition of the wire at each bend until fracture is indicated. B. Detached coating of a Ti-Nb wire with 200°C PTFE coating. Photo showing the condition of a 200°C coated Ti-Nb wire after seven bends.

Table 4 Number of bends until fracture of PTFE-coated bendable wires

| Material | PTFE coating (°C) | Number of bends until fracture | Coating detachment |
|----------|------------------|-------------------------------|-------------------|
| SUS      | Non              | 6.5 (0.55)                    | ND                |
|          | 200              | 6.3 (0.52)                    | ND                |
|          | 380              | 5.5 (0.55)                    | ND                |
| Ti-Mo    | Non              | 6.2 (0.41)                    | ND                |
|          | 200              | 4.7 (0.52)                    | ND                |
|          | 380              | 3.8 (0.41)                    | ND                |
| Ti-Nb    | Non              | 8.0 (0.63)                    | ND                |
|          | 200              | 7.5 (0.55)                    | 6.5 (0.55)        |
|          | 380              | 0 (0)                         | ND                |

n=6 for each experiment. Values are mean and (S.D.).

200°C heated Ti-Nb wires; however, the XRD angle of all the peaks were 1° greater with a small peak at 79° (Fig. 7 C-2).

The friction of PTFE is low. In this study, PTFE was coated only on the labial/buccal surfaces of rectangular straight wires (not arch form wires), so that the PTFE-coated side was attached by the elastic module and the other three metallic surfaces were attached to the wire slot surfaces of an edgewise bracket. Estimation of the friction between the PTFE-coated wires and a bracket revealed that PTFE-coating lowered the bracket–wire friction regardless of the type of wire, and the friction was lower than for non-coated wires (Table 5).

In the detachment test (Fig. 8), we checked and numbered detached parts of the PTFE coating on wires after toothbrushing under a stereoscopic microscope (Table 6). In all types of wires at all coating temperatures, the PTFE coating was detached by toothbrushing, and traces of abrasion could be observed (Figs. 8B, C and Table 6). Detachment of the coating was observed at the edge of coating, but not in the central portion, and abrasion traces were observed on the coating (Figs. 8B, C). No differences were observed in detachment between the 200°C and 380°C PTFE coating (Table 6).

In the wear test, we first tried to measure the worn volume of the PTFE-coating on the wires; however, this was difficult to evaluate. Instead, a wear test of PTFE-coated SUS plates was performed, and we then tried to estimate their worn depth. Although the toothbrushed portion and the boundary lines between intact and toothbrushed surfaces of the PTFE coating could be observed clearly with the naked eye and with a scanning electron microscope (Figs. 9A, B), the cross-sectional shapes and surface roughness of the PTFE surface scanned by a 3D laser measuring microscope had no discernible boundary lines and it was not possible to distinguish between intact and toothbrushed surfaces in non-coated, 200°C, and 380°C PTFE-coated SUS plates and orthodontic wires (data not shown).

The thickness of both the 200°C and 380°C PTFE coating was approximately 30–45 µm. The 380°C PTFE-coating was translucent, and appeared transparent over the color of the base metallic material. However, the 200°C PTFE-coating was able to mask the color of
Fig. 7 XRD profiles of orthodontic wires being heated during PTFE coating. 
A. SUS wires, B. Ni-Ti wires, C-1. Ti-Mo-Zr-Sn wires, C-2. Ti-Nb-Ta-Zr-O wires. Non-coated wires are intact products without coating or heating. ● indicates the austenite phase and ▼ indicates the martensitic phase.

Fig. 8 PTFE-coated wires after toothbrushing. 
A. Toothbrush with a strain gauge used in this study, B. Brushed surface of PTFE-coating on orthodontic wires, C. Scanning electron micrographs of abrasion traces and detached portions of PTFE-coated surfaces on orthodontic wires after toothbrushing. White and black lines represent 100 and 50 µm, respectively.

Fig. 9 PTFE-coated surfaces on SUS plates after the toothbrushing wear test. 
A. Stereoscopic micrographs of abrasion traces on PTFE-coated surfaces, B. Scanning electron micrographs of PTFE-coated surfaces, a) Non-coated, b) 200°C PTFE coating, and c) 380°C PTFE coating. Non-coated plates are intact SUS plates without coating or heating. White and black lines represent 500 µm.
Table 5  Friction between a bracket and PTFE-coated rectangular straight wires

| Material  | PTFE coating (°C) | Friction (gf)     |
|-----------|------------------|-------------------|
| SUS       | Non              | 123.94 \(\pm\) 10.367 |
|           | 200              | 102.98 \(\pm\) 7.020  |
|           | 380              | 103.17 \(\pm\) 7.476  |
| Ti-Mo     | Non              | 145.82 \(\pm\) 7.933  |
|           | 200              | 119.74 \(\pm\) 8.828  |
|           | 380              | 123.52 \(\pm\) 7.868  |
| Ti-Nb     | Non              | 152.61 \(\pm\) 10.145 |
|           | 200              | 123.42 \(\pm\) 9.638  |
|           | 380              | 124.40 \(\pm\) 8.496  |

\(n=6\) for each experiment. Values are mean and (S.D.).

Ti-Mo; Ti-11.5Mo-6Zr-4.5Sn, Ti-Nb; Ti-23Nb-0.7Ta-2.0Zr-1.2O, Non; intact products without PTFE coating or heating

Superscript letters denote statistically significant differences \((p<0.01)\) within each material.

Table 6  Estimation by stereoscopic microscopy of the detached portions of PTFE-coating on coated wires after toothbrushing

| Material  | PTFE coating (°C) | Detached portion no. |
|-----------|------------------|----------------------|
| SUS       | 200              | 0.5 \(\pm\) 0.42     |
|           | 380              | 1.0 \(\pm\) 0.63     |
| Ni-Ti     | 200              | 0.2 \(\pm\) 0.45     |
|           | 380              | 0.8 \(\pm\) 0.75     |
| β-titanium| 200              | 0.3 \(\pm\) 0.52     |
|           | 380              | 0.7 \(\pm\) 0.82     |
| Ti-Nb     | 200              | 0.2 \(\pm\) 0.41     |
|           | 380              | 0.7 \(\pm\) 0.52     |

\(n=6\) for each experiment. Values are mean and (S.D.).

Ti-Mo; Ti-11.5Mo-6Zr-4.5Sn, Ti-Nb; Ti-23Nb-0.7Ta-2.0Zr-1.2O

Superscript letters denote statistically significant differences \((p<0.01)\) within each material.

the base metallic material (Fig. 10). Coloration of the PTFE coating with food/drinks (curry, coffee, and tea) was examined and estimated as the color difference \((\Delta E)\) when compared with specimens immersed in DW (Fig. 10, Table 7). An experimental group of composite resin specimens was used as a positive control\(^4\). The PTFE-coated specimens exhibited low coloration after immersion in food and drinks, whereas the composite resin specimens were strongly colored. Similar to these results, bacterial adhesion of \(S.\) mutans and \(S.\) sanguinis to PTFE-coated surfaces was also lower than to non-coated SUS plates (Table 8).

**DISCUSSION**

PTFE, known as Teflon\(^6\), is a fluorocarbon solid, and is a high-molecular-weight compound consisting wholly of carbon and fluorine\(^7\). PTFE has inconsistent physical properties; it is a soft material, exhibiting low values for wear, friction, coloration, and bacterial adhesion\(^8\). In the medical and dental fields, it is used as a permanent and temporary graft material in surgical interventions.
Table 7  Color differences in specimens immersed in food and drink solutions

| Color difference ($\Delta E^{*ab}$) | Coffee | Tea | Curry |
|-----------------------------------|--------|-----|-------|
| Non coated                         | 3.51$^b$ (1.015) | 4.08$^b$ (0.897) | 4.26$^b$ (1.128) |
| 200°C PTFE coating                 | 1.27$^a$ (0.449) | 1.50$^a$ (0.419) | 1.99$^a$ (0.711) |
| 380°C PTFE coating                 | 1.14$^a$ (0.465) | 1.56$^a$ (0.273) | 2.01$^a$ (0.623) |
| Composite resin                    | 5.24$^b$ (1.121) | 5.73$^b$ (1.170) | 35.18$^a$ (2.434) |

$n=6$ for each experimental condition. Values are mean and (S.D.).

Color differences ($\Delta E^{*ab}$) of each specimen immersed in each drink/food solution were estimated by comparison with each material group immersed in distilled water.

Non-coated; intact SUS plates without PTFE coating or heating

Superscript letters denote statistically significant differences ($p<0.01$) for each drink/food.

Table 8  Bacteria adhesion of specimens

| PTFE-coating temperature (°C) | Bacterial adhesion test (% of SUS plate w/o PTFE coating) |
|-------------------------------|----------------------------------------------------------|
|                               | $S. mutans$ | $S. sanguinis$ |
| Non-coated                    | 100.00$^a$ (3.456) | 100.00$^a$ (4.528) |
| 200°C PTFE coating            | 30.56$^b$ (2.499) | 23.21$^b$ (1.653) |
| 380°C PTFE coating            | 24.33$^a$ (3.714) | 20.19$^a$ (1.136) |

$n=6$ for each experiment. Values are mean and (S.D.).

Non-coated; intact SUS plates without PTFE coating or heating

Superscript letters denote statistically significant differences ($p<0.01$) for each oral bacterium.

In recent times, orthodontic patients have tended to select clear or white-colored appliances. Esthetic orthodontic wires usually consist of metallic wires coated with white-colored resins, such as epoxy resin, polyurethane, and PTFE. PTFE is chemically stable, non-toxic to humans, highly resistant to heat and chemicals, and has an extremely low coefficient of friction. For these reasons, we performed this study investigating PTFE as a suitable coating material for orthodontic wires.

Titanium is a first transition series element, and is a dimorphic allotrope of an hexagonal α form that changes into a body-centered cubic (lattice) β form at 882°C. Titanium exhibits high osseointegration and biocompatibility; in other words, it is non-toxic, non-injurious to the body, and is not rejected by the body. It has many medical and dental uses, including surgical implants for joints and dental prostheses that can stay in the body for a long time. The high corrosion resistance of Ti is due to a passive film of titanium oxide (TiO$_2$) that forms on the surface under the existing environment of oxygen. Titanium alloys, such as nickel-titanium and β-titanium, exhibit special distinctive properties such as a lower modulus of elasticity (Young’s modulus) and superelasticity, so these alloys are widely used for orthodontic treatment. Despite these superior properties, titanium is originally a highly active metal that is easily oxidized at high temperatures. In the PTFE coating process, base materials are heated at temperatures above 200°C, which might change their mechanical properties. Heating especially at 380°C in Ti wires as well as SUS wires elevated their elastic modulus, which could be a kind of hardening heat treatment. In Ti-Nb wire, 200°C heating elevated its elastic modulus, which might be lowness of heat resistance of Nb. Conventional PTFE coating is performed at approximately 400°C, but low temperature-PTFE coating at 200°C has recently been established. Visual and stereoscopic microscopic observation has revealed dark-colored oxide films on β-titanium wires in particular after heating to 380°C (Fig. 3). SEM observation revealed that heating did not markedly change the surface condition of metal wires (Fig. 4). It is possible that the effects of low temperature coating on the mechanical properties of base materials are less marked than those of conventional high temperature coating.

After examining the transformation temperature and phase diagrams of titanium alloys, we first considered that both 200°C and 380°C heating for PTFE coating would not change the mechanical properties of base materials. The three-point bending test indicated that the mechanical strength of base wires of β-titanium
was affected by heating to 380°C, but not to 200°C, as evidenced by the elastic coefficient and maximum stress measurements (Table 3). Because Ni-Ti wires are usually heated to 400–500°C for a few minutes to a few hours to achieve shape fixity, we expected that 380°C heating would not alter their mechanical properties; however, we observed diminished superelasticity and permanent deformation in Ni-Ti wires heated to 380°C. Heating also increased the permanent deformation of Ti-Nb wires. The right-angle bending test showed that heating decreased the number of bends until fracture of all types of wires, especially at 380°C (Table 4), and the 380°C-heated Ti-Nb wires could not be bent to 90° at all. These results suggest that heating to 380°C might change the crystal structure phase of titanium wires, especially in Ni-Ti and Ti-Nb wires. Therefore, we performed XRD analysis of the heated wires.

In the SUS wires used in this study (SUS304; face-centered cubic lattice), the XRD profile was not altered by heating during PTFE coating. In contrast with intact SUS powder, each SUS wire exhibited a deformation-induced martensitic phase as a body-centered cubic structure diffraction peak at a 45° diffraction angle as well as an austenite phase as the parent phase at a 43° diffraction angle (Fig. 7A). These were probably caused by the deformation of the wire drawing and quenching processes.

For Ni-Ti wires (Ni-Ti; CaCl type-B2 structure), we first expected that the heating condition of the PTFE coating (200–400°C) would not change the crystal structure or the mechanical properties, based on the equilibrium diagram of Ni-Ti, and because Ni-Ti wires were usually heated at 400–500°C for a few minutes to a few hours to achieve shape fixity. Ni-Ti wire heated to 380°C exhibited a martensitic phase instead of an austenite phase at a 42° diffraction angle in unheated and 200°C heated specimens, which could be a cause of the diminishment of its superelasticity as revealed by the three-point bending test (Fig. 7B). The crystal structure of β-titanium exhibited a body-centered cubic structure. XRD profiles of 380°C heated Ti-Mo wire exhibited an almost identical pattern as unheated and 200°C heated Ti-Mo wires; however, the diffraction peaks of the 380°C heated Ti-Mo wires were markedly higher, especially the peak at a 56° diffraction angle (Fig. 7C-1). The profile of the 380°C heated Ti-Mo wire was closer to that of intact Ti-Mo powder than those of unheated and 200°C heated Ti-Mo wires. The weakened profiles of unheated and 200°C heated Ti-Mo wires might have been caused by the deformation of the wire drawing and quenching processes. Only the peak at 70°(6[211]) of the 380°C heated wires was 0.5° smaller than those of unheated and 200°C heated wires. A large surface separation of the crystals is required for θ to become small; however, the peak shift was observed only at 2θ=70°. In Ti-Mo wires, the mechanical properties were not markedly altered by 380°C heating, so the effects of this shift in the diffraction profile was unclear. In contrast with Ti-Mo wires, 380°C heating markedly reduced the mechanical strength of Ti-Nb wires. XRD profiles of 380°C heated Ti-Nb wire followed almost the same pattern as non-heated and 200°C-heated Ti-Nb wires; however, the XRD angle of all the peaks were 1° bigger (Fig. 7 C-2), which indicated that the surface separation of the crystals had increased. A heat-induced ω phase is easily formed at 373–573 K in β-titanium, which makes it harder and more fragile (known as ω brittleness). We suspect that the ω phase of 380°C heated Ti-Mo wire caused a marked reduction in its mechanical properties, so we repeated the XRD.

We found that a weak peak at 79° was detected at each of the six repeated XRD profiles, indicating a possible ω phase (Fig. 7 C-2). If this is the case, the change in the crystal structure might have caused the reduction in the mechanical strength of Ti-Nb wires by 380°C heating. Further study is needed to confirm the existence of the ω phase in Ti-Nb wires heated to 380°C. These results suggest that low temperature (200°C) PTFE-coating is a useful method that does not change the mechanical properties of base materials.

To clarify the clinical merits and disadvantages of PTFE-coated orthodontic wires apart from the mechanical properties, we evaluated the amount of friction between brackets and wires, the degree of wear and detachment of the coating caused by toothbrushing, the amount of coloration by foods and drinks, and the degree of microbial adhesion by oral bacteria.

The film thickness of 200°C and 380°C PTFE-coated wires in this study was approximately 30–45 µm (=0.0012–0.0018 inch), which is thicker than that of resin-coated white wires on the market, i.e. 25 µm (=0.0010 inch, numerical value on the catalogue). The surface roughness of the 200°C PTFE coating was coarser than that of the 380°C coating, which could be attributed to the coating method. The 200°C coating did not use a primer under the coating (Fig. 2). In this study, the PTFE was coated only on the labial/buccal side of the wires, so that wire width (i.e. the distance from the labial/buccal to the palatal/lingual sides) was increased by the film thickness of the PTFE coating (30–45 µm). This increased width of wire was predicted to increase the friction between the brackets and the wires; however, the PTFE-wires exhibited lower friction than non-coated wires by 20–30 gf (Table 5). This indicated that the surface roughness and morphology of PTFE coating might be hardly influenced the friction between wires and brackets. The low friction characteristics of PTFE should lower the friction of PTFE-coated wires against elastic modules directly touching the wire surfaces.

The results of the wear test with toothbrushing indicated that detachment of the coating was minimal, could not be distinguished with direct observation, and was not dependent on the base material. We also tried to estimate the amount of wear caused by toothbrushing; however, the boundary lines and the surface roughness between intact (non-worn) and worn surfaces on the PTFE coating could not be evaluated by a 3D laser measuring microscope (data not shown). Additionally, the worn surface could be distinguished by direct observation and with a scanning electron microscope.
wires, especially for titanium wires. Making it an excellent coating method for orthodontic low temperature (200°C) PTFE coating does not alter dental implants and artificial joints, could provide better friction, rather than surface roughness. These results suggest that PTFE-coating for orthodontic wires and other dental-medical devices, such as abutments for dental implants and artificial joints, could provide better function, esthetics, and oral hygiene.

From the results of this research, we conclude that low temperature (200°C) PTFE coating does not alter the mechanical properties of base metallic materials, making it an excellent coating method for orthodontic wires, especially for titanium wires.

CONCLUSION

The results of the current study show that low temperature (200°C) PTFE coating improves the metallic appearance of orthodontic wires, especially Ni-Ti and Ti-Nb wires, without changing their mechanical properties as determined by observation of crystal structure changes by XRD analysis. Wires with either conventional (380°C) or low temperature (200°C) PTFE coating both exhibited low coloration, low microbial adhesion, low friction against metallic brackets, and high tolerance of detachment and wear against toothbrushing.

We conclude that low temperature (200°C) PTFE coating is an excellent method for improving the appearance of orthodontic wires, especially titanium wires.

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CONFLICT OF INTEREST

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