Original Article

Effect of thermocycling-induced stress on properties of orthodontic NiTi wires

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Received 9 July 2022; Final revision received 22 July 2022
Available online 16 August 2022

KEYWORDS
NiTi wire; bending stress; Temperature fluctuation; Stress hysteresis; Crystallinity

\textit{Background/purpose:} In orthodontic applications, NiTi wires are under continuous bending stress and exposed to fluctuations in temperature over long durations. The sensitivity of NiTi to temperature can have a considerable influence on its mechanical properties. This study investigated the effects of deflected NiTi wire, presented in stress-induced (detwinned) martensite microstructure, combined with thermal cycle on the microstructure and mechanical properties.

\textit{Materials and methods:} We tested four types of as-received orthodontic NiTi: (1) Nitinol Classic (3 M Unitek), (2) Sentalloy (Tomy), (3) 27\textdegree C CuNiTi (Ormco) and (4) 40\textdegree C CuNiTi (Ormco). Each group of specimens was subjected to three different testing conditions: (1) temperature fluctuations (5000 cycles) between 5 and 55\textdegree C, (2) continuous three-point bending force and (3) combination of thermal cycling and bending stress.

\textit{Results:} The specimens that underwent thermocycling as well as loading exhibited a substantial narrowing in stress hysteresis, which may be attributed to crystallinity lower than that of as-received NiTi wires. Reduced crystallinity can manifest in a number of imperfections, such as dislocations and internal stress, as well as a less-organized structure. Micro X-ray diffraction (XRD) analysis revealed the existence of martensite phase in Sentalloy wires subject to thermal and stress conditions. Under loading conditions, stress-induced martensite of NiTi wires exposed to temperature fluctuations of 5–55 \textdegree C also induced cyclic changes in bending stress. In a simulated intra-oral environment, the stability of austenite→martensite transformation decreased.

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https://doi.org/10.1016/j.jds.2022.07.017
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Introduction

Nickel-titanium (NiTi) wires are widely used in orthodontics, due to their shape memory and superelasticity, which provide moderate, continuous force to induce tooth movement in the initial leveling and alignment stage. The shape memory and superelasticity is partly due to the crystallographically reversible nature of the displacive (diffusionless) martensitic transformation induced by changes in temperature or applied stress. Throughout the course of orthodontic treatment, NiTi wires are in place for time frames in the order of 1 year, such that the long-term microstructure and mechanical behavior of NiTi wires largely determines their clinical performance. The continuous bending (loading) conditions and exposure to temperature fluctuations (variations) associated with eating and drinking hot and cold items can alter the properties of NiTi wires. Barwart determined that even minimal changes in temperature can have a greater effect on the force delivery of superelastic NiTi springs than on stainless steel coil springs. In one in vivo study, the heating enthalphy of as-received 27 °C CuNiTi archwires is far below that of devices that had been used in patients for three months. Bourauel et al. claimed that NiTi wires react differently to variations in temperature. From these results, we can infer that in vivo variations in temperature and bending stress may degrade the mechanical properties of NiTi wires.

NiTi alloys are found in three microstructural phases: austenitic, martensitic, and intermediate rhombohedral or R-phase. Upon loading, austenite and twinned martensite undergoes elastic deformation, leading to a transformation into de-twinned martensite above a critical slip stress level. The deflected NiTi wire used in dental clinical applications presents a stress-induced (detwinned) martensite microstructure, such that any increase in temperature cavity increases stress. A number of studies have investigated the effects of thermocycling on the phase transformations and mechanical properties of NiTi wires. Other studies have evaluated the effects of long-term repeated deflections on mechanical properties. However, when evaluating the long-term properties of NiTi wires, those studies did not simulate actual orthodontic conditions. In clinical applications, temperature fluctuations can alter the stress in loaded NiTi wires. This study simulated a realistic intra-oral environment to investigate the effects of repeated temperature fluctuations and continuous three-point bending stress on the microstructure and mechanical properties of orthodontic NiTi wires in vitro. We tested four types of as-received orthodontic NiTi: (1) Nitinol Classic (3 M Unitek), (2) Sentalloy (Tomy), (3) 27 °C CuNiTi (Ormco) and (4) 40 °C CuNiTi (Ormco). Nitinol Classic wire belongs to work-hardening structure without temperature-induced phase transformation and shape memory effect. Sentalloy wire belongs to active austenitic grain structure with constant force over a long range of activation and temperature-induced phase transformation, but the temperature is lower than oral temperature. 27 °C CuNiTi and 40 °C CuNiTi wire belong to active austenitic grain structure with low stress–strain hysteresis and temperature-induced phase transformation, and the temperature could be accurately controlled in or near oral temperature.

Materials and methods

Materials

This study employed four types of commercial NiTi orthodontic archwire, with nominal cross-section dimensions of 0.016 × 0.022 inch. The wires were Nitinol Classic (3 M Unitek, Monrovia, CA, USA), Sentalloy (Tomy International, Tokyo, Japan), 27 °C copper (Ormco Corp., Glendora, CA, USA) and 40 °C copper NiTi (Ormco Corp.). The chemical composition of the four tested materials was analyzed using a scanning electron microscope (SEM) (JSM-6390 LV; JEOL, Tokyo, Japan) equipped with an energy dispersive X-ray spectrometer (INCA 350; Oxford Instruments, High Wycombe, UK). The chemical composition of the wires (in wt%) was as follows: as-received Nitinol Classic (47.9% titanium and 52.1% nickel) for wires; as-received Sentalloy (48.6% titanium and 51.4% nickel); 27 °C copper NiTi (47.86% titanium, 48.24% nickel, 3.83% copper, 0.08% chromium), and 40 °C copper NiTi (46.69% titanium, 48.31% nickel, 4.65% copper, 0.35%). The phase transformation temperature (PTT) of the four tested materials was analyzed using a differential scanning calorimetry experiments (DSC 200 F3 Maia®, Netzche, Selb, Germany). No phase transformation was observed in Nitinol Classic wires during heating and cooling processes. The PTT values of Sentalloy wires were as follows: (a) heating stage: R phase-start temperature (R notation) (4.9 °C) and austenite-finish temperature (A notation) (25.6 °C); (b) cooling stage: R (19.3 °C), R phase-finish temperature (R notation) (12.3 °C), martensite-start temperature (M notation) (–31.2 °C) and martensite-finish temperature (M notation) (–87.5 °C). The PPT values of 27 °C CuNiTi wires were as follows: (a) heating stage: austenite-start temperature (A notation) (4.1 °C) and A (24.4 °C); (b) cooling stage: M (6.4 °C) and M (–22.5 °C). The PPT values of 40 °C CuNiTi wires were as follows: (a) heating stage: A (13.9 °C)
and \( A_r \) (32.0 °C); (b) cooling stage: \( M_s \) (10.9 °C) and \( M_f \) (–8.9 °C). A 30-mm test specimen (free from induced internal stress) was sectioned from the end of an archwire using a water-cooled diamond-embedded saw (Isomet, Buehler, Lake Bluff, IL, USA).

Simulated intra-oral treatment

The as-received NiTi wires were first cleaned in 95% alcohol and rinsed using double distilled water. The wires were then divided into four groups for treatment. T group: Samples were placed in a tea strainer and thermally cycled between 5 and 55 °C for 5000 cycles.\(^{10,11}\) The dwell time in each batch was 90 s, and the transfer time was 15 s. S group: NiTi wires were exposed to continuous mechanical stress mimicking the intraoral conditions found in orthodontic applications. A three-point flexure fixture was used for the application of bending force. The device was moved a distance of 3.0 mm over a span of 13 mm, from the distal end of the bracket (canine tooth) to the mesial end of the bracket (second premolar). The flexure fixture holding a NiTi wire was placed in an oven at 37 °C for a duration equivalent to 5000 thermal cycles. \( T + S \) group: NiTi wires were fixed in a three point flexure fixture and exposed to bending load while undergoing 5000 thermocycles at temperatures between 5 °C and 55 °C. Following treatment, the specimens were removed from the flexing device, cleaned in 95% alcohol, rinsed in double distilled water, and then dried.

Micro X-ray diffraction (Micro-XRD) analysis

The phase composition of the wires was identified using micro X-ray diffractometry (Bruker AXS Gmbh, Karlsruhe, Germany) with Cu K\( _\alpha \) radiation at 40 kV and 40 mA. The sampling stage was oscillated from 30° to 100° (2\( \theta \)). After the wire was placed on the sample stage, the stage was carefully adjusted to the center portion of the wire.

Figure 1  Micro-XRD patterns at 37 °C using diffraction angles from 30° to 100° in four types of NiTi wire: (a) Nitinol Classic; (b) Sentalloy; (c) 27°C CuNiTi; (d) 40°C CuNiTi. Thermal cycle: samples were thermal cycled for 5000 cycles; Stress: samples fixed in 3-point flexure fixture were exposed to continuous bending stress; Thermal cycle + Stress: samples were exposed to continuous bending stress and thermocycled for 5000 cycles.
specimen. The micro-XRD pattern was obtained at 37 °C in a temperature-controlled chamber. The crystallinity of the austenite was evaluated according to the relative index of crystallinity (IOC), as defined by the ratio of the main peak (110) intensity of the treated wire (I_t) and the as-received wire (I_a) according to the following relationship: IOC (%) = (I_t/I_a) 100%. This method assumes that the IOC of the as-received wire is 100%.

### Table 1 Relative index of crystallinity (IOC) calculated from X-ray diffraction (XRD) patterns from as-receive and treated NiTi orthodontic wires.

| Control Thermal cycle | Bending stress | Thermal cycle + Bending stress |
|-----------------------|----------------|-------------------------------|
| Nitinol classic       | 100%           | 93%                           | 86%                           | 74%                           |
| Sentalloy             | 100%           | 94%                           | 94%                           | 46%                           |
| 27 °C CuNiTi          | 100%           | 100%                          | 75%                           | 45%                           |
| 40 °C CuNiTi          | 100%           | 97%                           | 87%                           | 77%                           |

Note: This method assumes that the IOC of the as-received wire is 100%.

### Three-point bending test

Following the simulation of intra-oral treatment, three-point bending tests were conducted using a universal testing machine (Autograph AGS-J, Shimadzu Co., Japan) in a temperature-controlled chamber. The span length between the two supports was 13 mm, with the center portion connected to a load cell (50 N). Bending load was applied to the center of the wire specimen at a rate of 1.0 mm/min, which resulted in a maximum deflection of 3.0 mm (loading process). The load was subsequently removed at the same rate (unloading process). The three-point bending test was conducted at 37 °C. As shown in load-deflection curves, Load A is martensite transformation start point; Load B is finish point during loading process; Load C is start point during unloading process; Load D is austenite re-transformation start point; Load E is austenite re-transformation finish point.

### Statistical analysis

At least three samples were analyzed for each time point, with results presented as mean ± standard deviation (SD).

![Load-deflection curves at 37 °C in four types of NiTi wire](image)

Figure 2  Load-deflection curves at 37 °C in four types of NiTi wire: (a) Nitinol Classic; (b) Sentalloy; (c) 27 °C CuNiTi; (d) 40 °C CuNiTi. Thermal cycle: samples were thermocycled for 5000 cycles; Stress: samples fixed in 3-point flexure fixture were exposed to continuous bending stress; Thermal cycle + Stress: samples were exposed to continuous bending stress and thermocycled for 5000 cycles.
The results were analyzed according to one-way analysis of variance (ANOVA) and the Duncan test ($P < 0.05$) using Statistical Analysis System software (SAS; SAS Institute Inc., Cary, NC, USA)).

**Results**

As shown in Fig. 1, XRD diffractograms at 37 °C were obtained from Nitinol Classic, Sentalloy, 27°C-CuNiTi, and 40°C-CuNiTi wires that had undergone various treatments. The as-received Nitinol Classic wire presented 110, 200, 211, and 220 peaks associated with austenite. Following simulated intra-oral treatment, the Nitinol Classic NiTi wires presented the original austenitic microstructure in all respects except for crystallinity. Table 1 presents the relative index of crystallinity (IOC) as follows: T group (93%), S group (86%), and T + S group (74%). Thermal cycling between 5 °C and 55 °C also altered the microstructure of the Nitinol Classic NiTi wires. The combination of thermal cycling and stress treatment greatly reduced the crystallinity of the specimens. Among the Sentalloy wires, the T + S group presented a decrease in the intensity of the main austenite peak and the appearance of a 020 martensite peak after thermal cycling and loading treatment. As shown in Table 1, the Sentalloy wires presented the following IOC values at 37 °C: T group (94%), S group (94%), and T + S group (46%). The XRD results of 27 °C CuNiTi wires presented the 110, 200, 211, and 220 peaks associated with austenite. Following T + S treatments, stress-deflection (mm) 37 °C CuNiTi wires reduced the crystallinity to 55%. The application of thermal cycling and stress treatment greatly affected crystallinity at 37 °C. The XRD results indicate the existence of austenite in the four 40 °C CuNiTi wires. The IOC values of 40 °C CuNiTi wires were as follows: T group (97%), S group (87%), and T + S group (77%). The application of thermal cycling and stress treatment to the 40 °C CuNiTi wires reduced the crystallinity to below that of the T-treated and S-treated groups.

Table 2 presents the stress-deflection curves obtained at 37 °C in NiTi wires following various treatments. Table 2 presents a summary of stress and residual deflection data obtained from the stress-deflection curves. We obtained the hysteresis loop of the stress-deflection curve in as-received, T-treated, S-treated, and T + S-treated Nitinol Classic NiTi wires. Under loading, S and T + S groups presented significantly lower stress versus deflection. Applied stress (Load B) at 3.0-mm deflection can be ranked as follows in decreasing order: as-received group/T group, S group, T + S group ($P < 0.05$). During unloading, the T + S group required greater stress for deflection. The stress (Load C) at the start point during the unloading process can be statistically ranked as follows in increasing order: as-received group/T group/S group, T + S group. After 3-point testing, T + S-treated specimens presented significantly larger residual deflection than did the other specimens. The as-received, T-treated and S-treated Sentalloy specimens presented good superelastic behavior with good deformation recovery and the formation of the typical flag-shaped hysteretic loop. Following T + S treatments, stress-deflection at 37 °C presented a low slope of stress versus deflection under initial loading conditions and residual deflection after unloading. Among the Sentalloy wires, the T-treated and T + S-treated groups presented narrow hysteresis loop during 3-point bending test. As shown in Table 2, the stress (Load A) was measured at the beginning of the

| Wire               | Group | Load A (MPa) | Load B (MPa) | Load C (MPa) | Load D (MPa) | Load E (MPa) | Plastic deformation (mm) |
|--------------------|-------|--------------|--------------|--------------|--------------|--------------|--------------------------|
| Nitinol classic    | Control | 692.4<sup>a</sup> | 403.4<sup>b</sup> | 403.4<sup>b</sup> | --           | 0.06<sup>b</sup> | --                       |
|                   | T      | 693.7<sup>a</sup> | 413.3<sup>b</sup> | 413.3<sup>b</sup> | --           | 0.06<sup>b</sup> | --                       |
|                   | S      | 625.6<sup>b</sup> | 399.4<sup>b</sup> | 399.4<sup>b</sup> | --           | 0.09<sup>b</sup> | --                       |
|                   | T + S  | 603.1<sup>c</sup> | 435.9<sup>a</sup> | 435.9<sup>a</sup> | --           | 0.15<sup>a</sup> | --                       |
| Sentalloy          | Control | 285.7<sup>a</sup> | 370.5<sup>a</sup> | 226.3<sup>b</sup> | 106.4<sup>b</sup> | 104.5<sup>a</sup> | --                       |
|                   | T      | 275.0<sup>ab</sup> | 360.6<sup>b</sup> | 219.2<sup>b</sup> | 115.6<sup>b</sup> | 109.7<sup>a</sup> | --                       |
|                   | S      | 269.1<sup>b</sup> | 376.9<sup>a</sup> | 249.2<sup>a</sup> | 107.3<sup>b</sup> | 105.8<sup>a</sup> | --                       |
|                   | T + S  | 246.2<sup>c</sup> | 358.6<sup>b</sup> | 252.1<sup>a</sup> | 126.8<sup>a</sup> | 113.2<sup>a</sup> | 0.21                     |
| 27°C CuNiTi        | Control | 281.3<sup>a</sup> | 394.9<sup>a</sup> | 257.3<sup>b</sup> | 173.1<sup>b</sup> | 173.6<sup>ab</sup> | --                       |
|                   | T      | 278.0<sup>a</sup> | 385.7<sup>ab</sup> | 245.2<sup>bc</sup> | 171.6<sup>b</sup> | 171.6<sup>ab</sup> | --                       |
|                   | S      | 276.9<sup>a</sup> | 395.4<sup>a</sup> | 240.3<sup>c</sup> | 170.7<sup>b</sup> | 177.0<sup>a</sup> | --                       |
|                   | T + S  | 245.7<sup>b</sup> | 382.8<sup>b</sup> | 280.7<sup>a</sup> | 186.3<sup>a</sup> | 167.2<sup>b</sup> | 0.09                     |
| 40°C CuNiTi        | Control | 230.1<sup>a</sup> | 314.5<sup>b</sup> | 213.5<sup>b</sup> | 79.5<sup>a</sup> | 108.7<sup>a</sup> | --                       |
|                   | T      | 236.1<sup>a</sup> | 327.8<sup>a</sup> | 184.2<sup>b</sup> | 97.0<sup>a</sup> | 110.2<sup>a</sup> | --                       |
|                   | S      | 225.3<sup>b</sup> | 298.4<sup>c</sup> | 174.8<sup>b</sup> | 92.2<sup>a</sup> | 99.0<sup>b</sup> | --                       |
|                   | T + S  | 218.9<sup>c</sup> | 315.0<sup>b</sup> | 196.2<sup>a</sup> | 96.1<sup>a</sup> | 104.8<sup>a</sup> | 0.07                     |

Notes: Number of samples = 3; Group Control: as received NiTi wires; Group T: samples were thermocycled for 5000 cycles; Group S: samples fixed in 3-point flexure fixture were exposed to continuous bending stress; Group T + S: samples were exposed to continuous bending stress and thermocycled for 5000 cycles; Load A: martensite transformation start point; Load B: finish point during loading process; Load C: start point during unloading process; Load D: austenite re-transformation start point; Load E: austenite re-transformation finish point; ANOVA and Duncan test ($P < 0.05$) were performed, and different scale-down letters on right for each group represent significant differences within each wire.
transformation of austenite into martensite. Load A of the T + S-treated specimen was significantly lower than that of the other groups. The stress (Load B) measured at 3.0-mm deflection under loading process can be ranked as follows in increasing order: as-received group/S-treated group, T-treated group/T + S-treated group. During unloading, the T + S group presented higher stress prior to the completion of the martensite → austenite transformation. After 3-point testing, the residual deflection was 0.21 mm in the T + S group. Among the 27 °C CuNiTi wires, the as-received group, T-treated group, and S-treated groups recovered entirely from deflected deformation. After thermal cycling and loading treatment, the stress-deflection curve of 27 °C CuNiTi wire presented a low slope under initial loading and residual deflection after unloading. Load A of the T + S-treated group was significantly lower than that of the other groups. Load C and Load D of the T + S-treated 27 °C CuNiTi wire was statistically higher than that of the other specimens. We observed a flag-shaped hysteresis loop in the stress-deflection behavior of the four 40 °C CuNiTi wires, whereas the T + S-treated specimen showed a low slope of stress versus deformation under initial loading conditions, permanent deformation after unloading, and a narrow hysteresis loop. As shown in Table 2, the T + S-treated group presented significantly lower Load A than did the other groups during loading, and the T + S group presented statistically higher Load C than did the other groups at the reverse point under unloading.

Discussion

The NiTi wires used for orthodontic therapy are generally under conditions of continuous loading and thermal fluctuation for prolonged periods (i.e., 1 year). NiTi is sensitive to fluctuations in mouth temperature, which can induce dynamic changes (variation) in applied stress. When under load, twinned martensite (low temperature) and austenite (high temperature) microstructures transform into a stress-induced (detwinned) martensite microstructure. A lack of research on the effects of thermal cycling and stress fluctuation on detwinned martensite has hindered attempts to predict clinical performance. Berzins and Roberts used differential scanning calorimetry (DSC) to detect changes in phase transformation in commercial NiTi wires thermocycled between 5 and 55 °C. They reported qualitative as well as quantitative changes in phase transformation under thermocycling, which can be attributed to an increase in the number of dislocations. Under the same thermocycling conditions, we were unable to detect changes in the flag-shaped hysteresis of 27 °C CuNiTi specimens. These mechanical properties are inconsistent with the results of DSC. This could be attributed to the fact that an increase in the number of dislocations may have no effect on the mechanical properties of NiTi wires at the temperatures encountered in the oral-cavity. The thermocycling treatment is not a good method to evaluate the properties of NiTi wires in orthodontic applications.

In the present study, we investigated the effects of thermal cycling and bending stress on three functional types of NiTi orthodontic wires. The function of NiTi orthodontic NiTi wires could be divided into three types: (1) Nitinol Classic with lower elastic modulus and higher elastic range, (2) Sentalloy and 27 °C CuNiTi of austenitic-active type with superelasticity, and (3) 40 °C CuNiTi of martensitic-active type with shape memory. As shown in Fig. 2 (a), T-treated Nitinol Classic wires present the same flag-shape hysteresis as as-received wire. This could be attributed to the fact that Nitinol Classic do not undergo phase transformation during heating and cooling. As shown in Fig. 2 (b) and (c), only thermocycled Sentalloy wires presented a narrow hysteresis loop in the stress-deflection
curves. The Sentalloy wires presented R phase at 5°C and austenite phase at 55°C. The R phase ↔ austenite phase transformation tends to alter thermal stability, which is related to microstructure and mechanical performance. The 27°C CuNiTi specimens presented mainly austenite during 5°C–55°C thermocycling. The thermal stability of 27°C CuNiTi results in steady mechanical properties. The 40°C CuNiTi wires presented a combination of austenite and martensite phases at 5°C and austenite phase at 55°C. The complexity of phase transformation decreases thermal stability, which alters the mechanical performance of 40°C CuNiTi.

As shown in Table 2, S-treated specimens must undergo constant stress at 37°C. The stress of Load C is 411.1 MPa in Nitinol Classic, 222.4 MPa in Sentalloy, 262.3 MPa in 27°C CuNiTi, and 181.5 MPa in 40°C CuNiTi. Higher stress values change the mechanical stability of NiTi wires. The S-treated Nitinol Classic wires present a narrow hysteresis loop in the stress–deflection curves. Bending stress (S treatment) was also shown not to have significant influence on the mechanical properties of the Sentalloy and 27°C CuNiTi wires in this study. This may be explained by the limited duration (about 12.2 day) of applied stress, which was equivalent to 5000 thermal cycles (i.e., in vivo exposure of approximately six months). If the applied stress were extended to the equivalent of six months of orthodontic treatment, the mechanical properties of S-treated NiTi wires may be altered.

The mechanical properties of NiTi are temperature sensitive.17 As shown in Fig. 3, three groups would induce different bending stress of 40°C CuNiTi wires in simulated intra-oral treatment. For T group, the 40°C CuNiTi wires would present the stress-free condition thermocycled between 5°C and 55°C. For S group, the 40°C CuNiTi wires were given 314 MPa at 37°C. For T + S group, the 40°C CuNiTi wires were kept at combination of temperature fluctuation between 5°C and 55°C and induced cycle loading between 162 MPa and 433 MPa. As shown in Fig. 4 (a) and (b), we used a 3-point bending test to obtain the values for Load B and Load C in as-received NiTi wires at 5°C, 37°C, and 55°C. The mechanical strength of NiTi wires increased with an increase in temperature. For example, the Load B of 40°C CuNiTi wires was 162.4 MPa at 5°C, 314.5 MPa at 37°C and 433.0 MPa at 55°C, respectively. As shown in Fig. 3, these results indicate that temperature fluctuations of 5°C–55°C would induce cyclic changes in bending stress during T + S treatment. The tension and relaxation of applied stress was shown to influence the mechanical stability of T + S-treated NiTi wires. Four types of NiTi wires presented low stress slope versus deflection under initial loading conditions, permanent deflection after unloading, and a narrow hysteresis loop after T + S treatment. The mechanical stability of austenite ↔ martensite transformation decreased in a simulated intra-oral environment.

Differences in the microstructure of NiTi wires subjected to thermal cycling and bending stress were evaluated using XRD. As shown in Table 2, the decrease in IOC could be attributed to the formation of imperfections, such as dislocations, internal stress, and precipitates, as well as a less-organized structure resulting from thermal cycling in conjunction with bending stress. Low crystallinity would tend to hinder phase transformations and eventually produce irreversible changes, such as residual plastic deformation. Pelton et al. have studied the effects of thermal cycling on microstructure and properties in Nitinol. They mentioned that the effect of thermal cycling requires the greater driving force for the martensitic transformation to overcome the resistive force due to these defects after thermal cycling.18 The low crystallinity of T + S-treated specimens presented significant changes in loading slope, residual deflection, and the narrow hysteresis loop in stress-deflection curves. For clinical application, it is very important to develop a new test method to investigate simulated intra-oral properties of NiTi. For example, our previous studies indicate that the bending stress would significantly change the corrosion resistance of NiTi wires.19,20

In conclusion, The composition and microstructure of NiTi wires were shown to determine long-term clinical performance under loading conditions. Orthodontic wire undergo dynamic conditions with regard to temperature and applied stress. It appears that this thermocycling treatment failed to simulate the actual conditions found in orthodontic applications. Stress hysteresis narrowing under thermal cycle and bending conditions, the clinical implication should be concerned about the force of the
unloading plateau is gradually increased. Researchers require a better understanding of the effects of temperature fluctuations on the mechanical properties of NiTi in order to advance the effectiveness of alloy wires in orthodontic applications.

Declaration of competing interest

The authors have no conflicts of interest relevant to this article.

Acknowledgments

This study was supported by the grant (MOST 109-2314-B-006-012-MY3) of Ministry of Science and Technology, Taiwan.

References

1. Barwart O. The effect of temperature change on, the load value of Japanese NiTi coil springs in the superelastic range. Am J Orthod Dentofacial Orthop 1996;110:553–8.
2. Sakima MT, Dalstra M, Melsen B. How does temperature influence the properties of rectangular nickel–titanium wires? Eur J Orthod 2006;28:282–91.
3. Biermann MC, Berzins DW, Bradley TG. Thermal analysis of as-received and clinically retrieved copper-nickel-titanium orthodontic archwires. Angle Orthod 2007;77:499–503.
4. Bourauel C, Scharold W, Jager A, et al. Fatigue failure of as-received and retrieved NiTi orthodontic archwires. Dent Mater 2008;24:1095–101.
5. Iijima M, Ohno H, Kawashima I, et al. Mechanical behavior at different temperatures and stresses for superelastic nickel-titanium wires with different transformation temperatures. Dent Mater 2002;18:88–93.
6. Berzins DW, Roberts HW. Phase transformation changes in thermocycled nickel-titanium orthodontic wires. Dent Mater 2010;26:666–74.
7. Es-Souni M, Es-Souni_M, Brandies HE. On the transformation behaviour, mechanical properties and biocompatibility of two NiTi-based shape memory alloys: NiTi42 and NiTi42Cu7. Biomaterials 2001;22:2153–61.
8. Urbina C, Flor SD, Ferrando F. Effect of thermal cycling on the thermomechanical behaviour of NiTi shape memory alloys. Mater Sci Eng A 2009;501:197–206.
9. Aken CAJM, Pallav P, Kleverlaan JK, et al. Effect of long-term repeated deflections on fatigue of preloaded superelastic nickel-titanium archwires. Am J Orthod Dentofacial Orthop 2008;133:269–76.
10. Moore RJ, Watts JT, Hood JA, et al. Intra-oral temperature variation over 24 hours. Eur J Orthod 1999;21:249–61.
11. International Organization for Standardization. ISO Technical Specification (ISO/TS) 11405: Dental materials-Testing of adhesion to tooth structure. 2003
12. Lee YH, Lim BS, Lee YK, et al. Comparison of transition temperature range and phase transformation behavior of nickel-titanium wires. Korean J Orthod 2010;40:40–9.
13. Mullins WS, Bagby MD, Norman TL. Mechanical behavior of thermo-responsive orthodontic archwires. Dent Mater 1996;12:308–14.
14. Thayer TA, Bagby MD, Moore RN, et al. X-ray diffraction of nitinol orthodontic arch wires. Am J Orthod Dentofacial Orthop 1995;107:604–12.
15. Miyazaki S, Sachdeva RL. Shape memory effect and superelasticity in Ti–Ni alloys. In: Yoneyama S, Miyazaki S, eds. Shape memory alloys for biomedical applications. Cambridge: Woodhead Publishing Limited, 2009:3–19.
16. Brantley WA. Orthodontic wires. In: Brantley WA, Eliades T, eds. Orthodontic materials: scientific and clinical aspects. New York: Thieme, 2001:77–103.
17. Meling TR, Odegaard J. The effect of short-term temperature changes on the mechanical properties of rectangular nickel titanium archwires tested in torsion. Angle Orthod 1998;68:369–76.
18. Peltona AR, Huangb GH, Moinec P, et al. Effects of thermal cycling on microstructure and properties in Nitinol. Mater Sci Eng 2012;532:130–8.
19. Liu JK, Lee TM, Liu IH. Effect of loading force on the dissolution behavior and surface properties of nickel-titanium orthodontic archwires in artificial saliva. Am J Orthod Dentofacial Orthop 2011;140:166–76.
20. Liu IH, Lee TM, Chang CY, et al. Effect of load deflection on corrosion behavior of NiTi wire. J Dent Res 2007;86:539–43.