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Abstract
Objectives: The purpose of this study was to investigate the suitability of the Bend and Free Recovery (BFR) method as a standard test method to determine the transformation temperatures of heat-activated Ni-Ti orthodontic archwires. This was done by determining the transformation temperatures of two brands of heat-activated Ni-Ti orthodontic archwires using the both the BFR method and the standard method of Differential Scanning Calorimetry (DSC). The values obtained from the two methods were compared with each other and to the manufacturer-listed values.
Methods: Forty heat-activated Ni-Ti archwires from both Rocky Mountain Orthodontics (RMO) and Opal Orthodontics (Opal) were tested using BFR and DSC. Round (0.016 inches) and rectangular (0.019 × 0.025 inches) archwires from each manufacturer were tested. The austenite start temperatures (A_s) and austenite finish temperatures (A_f) were recorded.
Results: For four of the eight test groups, the BFR method resulted in lower standard deviations than the DSC method, and, overall, the average standard deviation for BFR testing was slightly lower than for DSC testing. Statistically significant differences were seen between the transformation temperatures obtained from the BFR and DSC test methods. However, the A_f temperatures obtained from the two methods were remarkably similar with the mean differences ranging from 0.0 to 2.1 °C: A_f Opal round (BFR 26.7 °C, DSC 27.6 °C) and rectangular (BFR 27.6 °C, DSC 28.6 °C); A_f RMO round (BFR 25.5 °C, DSC 25.5 °C) and rectangular (BFR 28.0 °C, DSC 25.9 °C). Significant differences were observed between the manufacturer-listed transformation temperatures and those obtained with BFR and DSC testing for both manufacturers.
Significance: The results of this study suggest that the Bend and Free Recovery method is suitable as a standard method to evaluate the transformation temperatures of heat-activated Ni-Ti orthodontic archwires.
Keywords: Bend and Free Recovery, Differential Scanning Calorimetry, Recovery temperature testing apparatus, Nickel-Titanium, Shape memory,

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Heat-activated, Orthodontic archwires, Transformation temperatures, Austenite start temperature, Austenite finish temperature

1. Introduction

Within orthodontics, standards for the manufacturing of products provide distinct guidelines and clarity to manufacturers and consumers mutually.[1] Set standards that provide requirements for measurement and labeling of wire size, along with requirements for testing and presenting of physical and mechanical properties of orthodontic wires, have made the comparison between products easier for clinicians. However, many U.S. manufacturers do not provide packaging and labeling information required by ANSI/ADA and ISO standards for orthodontic wires. In particular, both ANSI/ADA Standard No. 32 "Orthodontic Wires" and ISO 15841 "Dentistry–Wires for use in Orthodontics" require that, when applicable, the austenite finish temperature (A_f) of nickel-titanium (NiTi) wires be provided with the packaging and labeling information.[2;3] Yet, information on the austenite finish temperature is often not found on the labels of orthodontic wires claiming to be "heat-activated."

Nickel-titanium alloys have the ability to exhibit a shape memory effect. The ASTM Committee F04 on Medical and Surgical Materials and Devices defines a "shape memory alloy" to be an alloy that, after it is plastically deformed in the martensitic phase, "undergoes a thermoelastic change in crystal structure when heated through its transformation temperature range resulting in a recovery of the deformation."[4] It is this shape memory effect exhibited by NiTi alloys that is used by the Bend and Free Recovery method to determine transformation temperature values, as described below. The high temperature phase for NiTi shape memory alloys (SMAs) is referred to as the austenitic phase, and the lower temperature phase is the martensitic phase.[4] When in the austenitic phase, NiTi has a body-centered cubic crystal structure, making it difficult to displace; however, when it is in the martensitic phase, it has a close-packed hexagonal crystal structure, which allows the molecules to slide across one another more easily.[5] The martensitic phase has a lower modulus of elasticity (~50 GPa) than the austenitic phase (~120 GPa), which essentially means the martensitic phase is more flexible.[6]

The temperature range at which NiTi changes between its two solid phases (martensite and austenite) is called the Transformation Temperature Range (TTR).[4] Both phases exist within this range in a dynamic equilibrium.[7] The austenite start temperature (A_s) is the temperature at which the martensitic phase starts to transform to the austenitic phase when the alloy is heated.[4] Once the temperature is equal to or greater than the austenite finish temperature (A_f), the wire is entirely in the austenitic phase. Above A_f, the archwires have the ability to exhibit superelastic behavior. The archwires must be above A_f for the "nonlinear recoverable deformation behavior" characteristic of superelasticity to take place.[4] This is because the behavior comes from the "stress-induced formation of martensite on loading and the spontaneous reversion of this crystal structure to austenite upon unloading."[4] As stated above, when the temperature is below A_s and the wire is in the martensitic phase, it is more flexible.[6] Thus, since the archwire will exhibit different behaviors whether it is below A_s or above A_f, the transformation temperature range is one of the most important features of a thermoelastic (heat-activated) wire. Moreover, these heat-activated wires are significantly more expensive than many other types of NiTi archwires available for purchase, so it important to clinicians that these wires actually transition at the claimed clinically relevant temperature.

The majority of published orthodontic studies use Differential Scanning Calorimetry (DSC) to test the transformation temperatures of orthodontic wires. Also, standards for orthodontic wires, specifically ANSI/ADA Standard No. 32 and ISO 1584, specify DSC as the method for determination of the austenite finish temperature (A_f) for orthodontic archwires.[2;3] However, for some manufacturers within the medical device industry, DSC is not the preferred test method for determination of the A_f of NiTi devices. The Bend and Free Recovery (BFR) method, as described in ASTM F 2082 "Standard Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery", is also used to test and verify the A_f temperature of medical products such as nitinol stents.[8;9] Both of these methods (DSC and BFR) are straightforward to perform, able to test small specimens, and are reproducible.[8] However, since the BFR method has the ability to test a finished medical product without sectioning, the results obtained from this method can be more clinically relevant. Furthermore, it is the only

method that utilizes the shape memory effect of NiTi wires during testing, as noted by ASTM F 2082:[9] "measurement of the specimen motion closely parallels many shape memory applications and provides a result that is applicable to the function of the material." Also, when NiTi wire is bent around a mandrel of a suitable radius of curvature to induce "an outer fiber strain level of 2–2.5%", ruggedness testing has shown that the effect of applied strain is not significant.[9;10] However, BFR allows higher strain levels to be applied if the product being tested is subjected to higher strain levels during clinical use and the researchers would like to simulate the higher levels during testing. Since increasing strain has been shown to shift transformation temperatures to higher levels, simulating clinical strain levels is important.[8] Additionally, the apparatus used for BFR testing is much more economical in comparison to the price of DSC equipment.

Given this information, the absence of the BFR method for the testing of heat-activated NiTi archwires within the orthodontic literature is surprising. Therefore, the purpose of this study was to investigate the suitability of the Bend and Free Recovery method as a standard test method to determine the transformation temperatures of heat-activated NiTi orthodontic archwires. This was done by determining the transformation temperatures of two brands of heat-activated NiTi orthodontic archwires using both the Bend and Free Recovery method and the standard method of Differential Scanning Calorimetry. The values obtained from the two methods were compared with each other and to the manufacturer-listed values.

2. Materials and methods

The experimental groups consisted of commercially available thermoelastic NiTi orthodontic archwires from two different manufacturers, Opal Orthodontics (Opal; South Jordan, UT, USA) and Rocky Mountain Orthodontics (RMO; Denver, CO, USA). Round archwires with a diameter of 0.016 inch (0.41 mm) were chosen, since these are commonly used in the initial leveling and aligning phase of orthodontic treatment. Rectangular archwires with dimensions of 0.019 inch × 0.025 inch (0.48 × 0.635 mm) were also tested, since many practitioners use such wires early in treatment. Manufacturers were asked to provide wires from two different lots: Opal round -

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258999 and 245990; Opal rectangular - 261376 and 258671; RMO round – F1111747 and F1202886; and RMO rectangular – F1204539 and F1209259. There were a total of eight groups, each comprised of 10 specimens from two different lots, which is double the sample size used for the precision and bias statements of ASTM F 2082-06 and ASTM F 2004-05. All specimens were stored at room temperature prior to testing.

2.1. Bend and Free Recovery (BFR) test method

The Bend and Free Recovery test method was performed using the Recovery Temperature Testing Apparatus (RTTA). This apparatus was built at the American Dental Association (Chicago, IL, USA) using the apparatus requirements set forth in ASTM F 2082-06.[9] Since a closed BFR testing system was not used, testing of specimens was randomized to account for the potential environmental differences within the laboratory at different test times. An outside participant numbered specimens 1 through 20 for each group. These numbers were then randomized using the randomization feature in Microsoft® Excel (Redmond, WA, USA) to determine the order of testing. To avoid cutting and grinding, which can cause cold working of the material that affects the transformation temperature,[10;11] and to use actual orthodontic archwire products with material volumes relevant to their clinical function, the wires were tested as received without being cut. The wires were tested at a consistent location along their straight portions, 15 mm from the end of each archwire.

In brief, to determine the transformation temperature of a specimen by BFR, ASTM standard F 2082 states that the specimen must be cooled "to its nominally fully martensitic phase," deformed, and heated back to its fully austenitic phase.[9] During the heating process, the specimen movement is monitored; therefore, specimen displacement can be plotted versus specimen temperature. From the temperature−displacement graph, the A_s and A_f of the specimen can be determined (Fig. 1).

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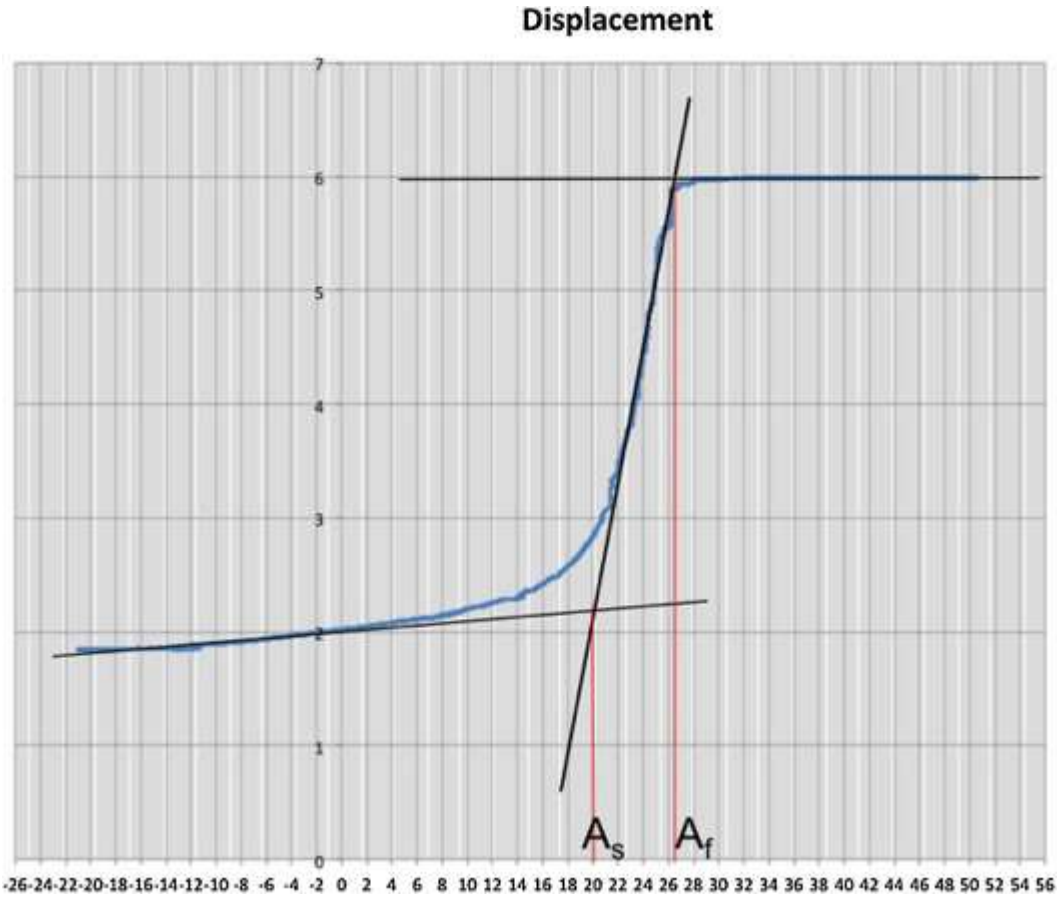

Fig. 1. Typical temperature–displacement graph to determine A_s and A_f for a one-stage transformation using the bend and free recovery test method. The *x*-axis is temperature in degrees Celsius, and the *y*-axis is displacement of the LVDT core in millimeters.

Before testing, each specimen was marked with a permanent marker 20 mm from one end. To begin a test, an individual wire was mounted on the test recovery fixture of the RTTA, with the wire clamped in position on the forming mandrel such that the 20 mm mark and the recovery fixture clamp were aligned, as shown in Fig. 2a. A bath was then filled with a water–glycerin solution that was cooled down to a minimum of -20 °C. Next, the test recovery fixture, with the test wire mounted on it, was placed in the water–glycerin bath, and a T-type thermocouple, with a resolution of 0.1 °C, was positioned as close as possible to the test wire (the thermocouple was calibrated by comparison with a NIST traceable, mercury reference thermometer with a resolution of 0.05 °C using a method similar to one described in ASTM E 220-02[12]). In order to allow the wire and RTTA parts to

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equilibrate to the bath temperature, the test wire remained in the water−glycerin solution for a minimum of 3 min prior to testing.

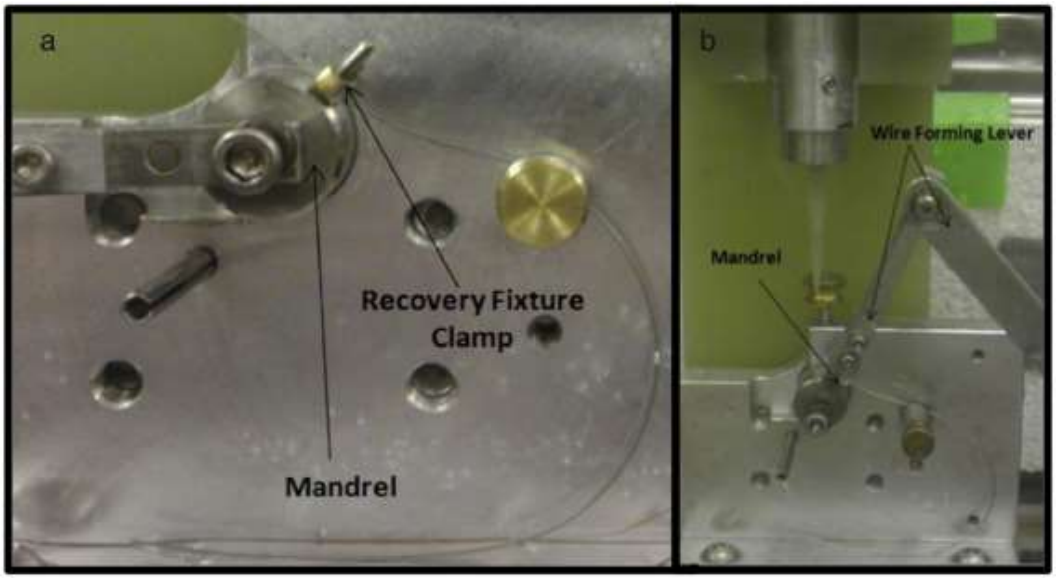

Fig. 2. (a,b). Recovery Temperature Testing Apparatus (RTTA). (a) Close-up of an individual wire mounted on the test recovery fixture of the RTTA, with the wire clamped in position on the forming mandrel such that the 20 mm mark and the recovery fixture clamp are aligned. (b) Close-up of the wire forming lever in position to be moved over a test wire, bending it against the forming mandrel.

After 3 min, the wire-forming lever was moved over the test wire, bending it against the forming mandrel (Fig. 2b). This wire deforming step resulted in the round wires being subjected to an outer surface strain of 2.5%, and the rectangular wires being subjected to a slightly higher outer surface strain of 2.95%. After the wire deformation step, the core of a linear variable displacement transducer (LVDT, Model DC 750-250-10, MacroSensors, Pennsauken, NJ, USA) was lowered onto the test wire 15 mm from the end. The LVDT specifications are the following: range ±6.3 mm, full-scale output 0 to ±10 V DC, and linearity error < ± 0.25% of full range output (note that the linearity was verified to be within specification using a procedure similar to the one outlined in ASTM F 2537).[13] The weight of the LVDT core was counterbalanced such that the weight on the test wire was no more than 3 g. Fig. 3 shows an illustration of the Recovery Temperature Testing Apparatus with the LVDT core lowered on to the test wire.

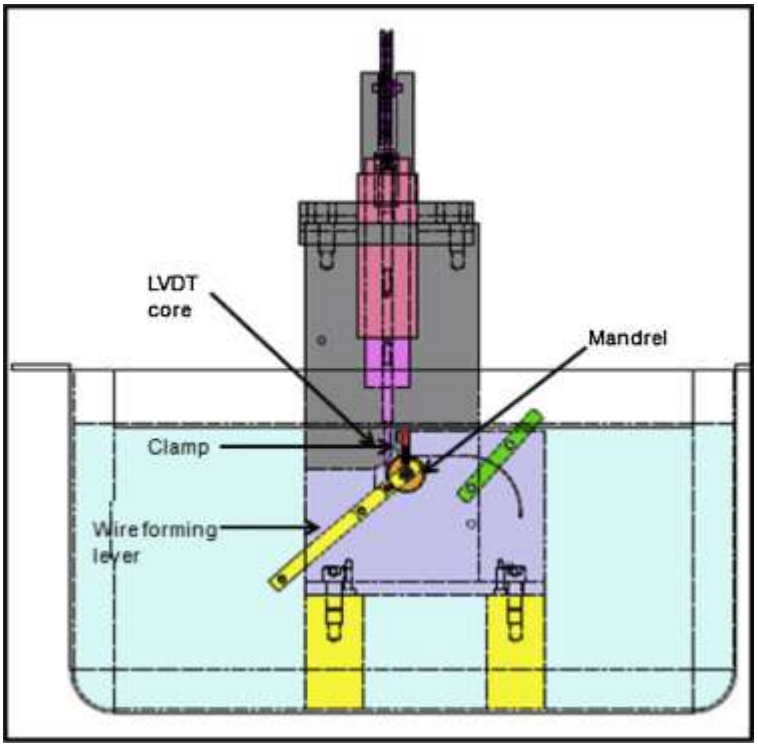

Fig. 3. Illustration of Recovery Temperature Testing Apparatus (RTTA) with different parts labeled and the linear variable displacement transducer core lowered on to a test wire (illustration provided by Henry Lukic of the American Dental Association).

After the LVDT core was positioned, a polyimide film insulated heater (Kapton® flexible heater, 10 W/in^2, Omega Engineering Inc., Stamford, CT, USA) was turned on to heat the water glycerin bath, and a stirrer was turned on to circulate the solution. The heating rate was limited to 1.4−1.6 °C/min. At the same time the heater was turned on, a data acquisition system (CompactDAQ, National Instruments Corp., Austin, TX, USA) was initiated to acquire the signals from the thermocouple and LVDT. From the acquired signals, temperature and displacement were monitored using a custom written program (LabVIEW software, National Instruments Corp.). For wires from both manufacturers, the tests were stopped at 50 °C, since this temperature was at least 10 °C above the A_f of both wire groups as determined by pilot testing.

The data from the data acquisition program were saved as text files and imported into a spreadsheet (Microsoft® Excel) for plotting. For each test, a temperature versus time graph was created to determine the heating rate for the individual test. Also, for each test, a

temperature versus displacement graph was created to determine A_s and A_f. This was done by using the spreadsheet tools to draw lines tangent to the different linear portions of an individual curve, in accordance with the procedure set forth in ASTM F 2082.[9] Fig. 1 shows a sample curve with the tangent lines drawn, and A_s and A_f determined by the intersection of the tangent lines.

2.2. Differential Scanning Calorimetry (DSC) test method

The DSC testing was performed using a Mettler Differential Scanning Calorimeter (Model 822e Mettler-Toledo Inc., Columbus, OH, USA). Specimen preparation included sectioning 5 mm segments from the straight portion end of each archwire using a low-speed, water-cooled diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA). For an individual test, a 5 mm segment was placed in an aluminum crucible and sealed (note that it was not necessary to bend the straight, 5 mm segment to fit it in the crucible). The test crucible and an empty aluminum crucible were placed in the differential scanning calorimeter at room temperature, and the temperature was scanned from -100 to 100 °C and back to -100 °C at a rate of 10 °C per minute. Liquid nitrogen was used as the coolant and nitrogen gas for purging.

The DSC plots were analyzed using the DSC manufacturer's software. A_s and A_f values were determined by the intersection of the baseline of the heating curve with tangents to the heating peak, as specified and illustrated in ANSI/ADA Standard No. 32 and ISO 15841.[2;3] Cooling peaks were also analyzed but were not included for comparison because the BFR did not record analogous values. Fig. 4 shows a representative DSC curve.

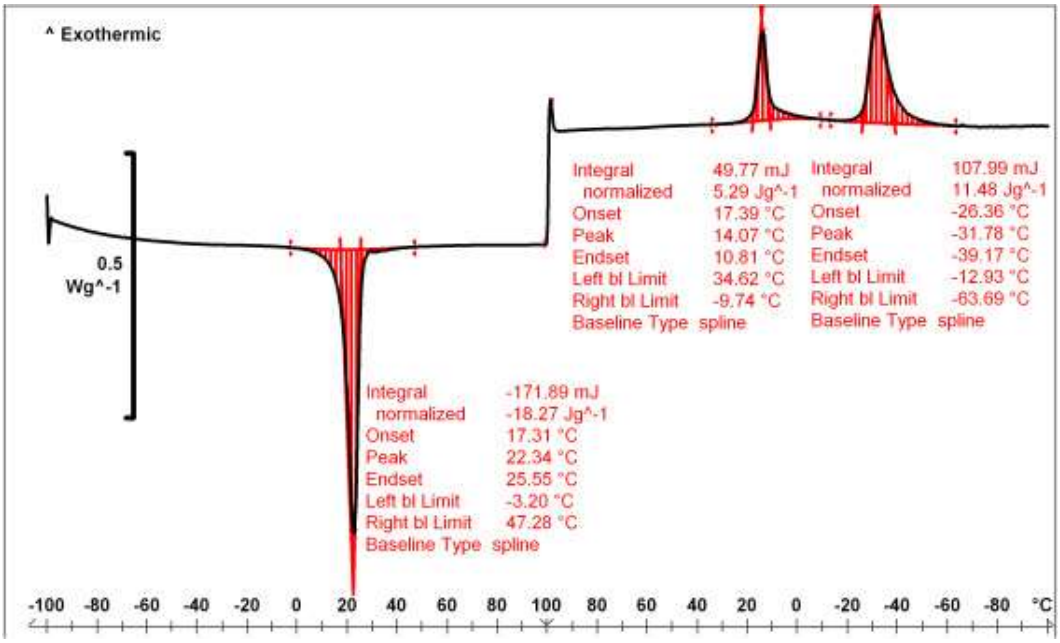

Fig. 4. Typical temperature versus time plot to determine A_s, A_f, M_s, and M_f using the differential scanning calorimetry method. The *x*-axis is temperature in degrees Celsius, and the *y*-axis is heat flow.

3. Statistical analysis

Mean A_s and A_f values were calculated for each wire group along with their respective ranges and standard deviations (Microsoft® Excel). Statistical analysis was performed on the data using Student's *t*-tests for two independent means. Independent samples *t*-tests were performed to make the following comparisons: transformation temperatures between the BFR and DSC test methods; between transformation temperature values of round and rectangular wires from the same manufacturer when tested using the BFR method; between transformation temperature values from the two manufacturers, Opal and RMO, when tested using the BFR method; between transformation temperature values of round and rectangular wires from the same manufacturer when tested using the DSC method; and between transformation temperature values from the two manufacturers, Opal and RMO, when tested using the DSC method. Also one sample *t*-tests were performed to compare the manufacturers' listed A_s and A_f values with the values from both the BFR and DSC tests. SPSS statistical software Version 19 (Chicago, IL, USA) was used for all statistical analysis.

4. Results

4.1. Comparison between BFR and DSC test methods

Statistically significant mean differences between the two test methods were seen for all A_s values, regardless of manufacturer or wire size ($p < 0.001$). Likewise, for the A_f values, statistically significant mean differences between the two test methods were seen for the A_f of round Opal wires (BFR 26.7, DSC 27.6, $p = 0.022$), and the A_f of both rectangular RMO (BFR 28.0, DSC 25.9, $p = 0.004$) and Opal (BFR 27.6, DSC 28.6, $p = 0.050$) wires, with the latter wires on the borderline of statistical significance (Table 1).

Table 1. Comparison of transformation temperatures between BFR and DSC test methods.

Wire size comparison	Variable by manufacturer		*N*	BFR $\bar{X}$ ± S.D. (°C)	DSC $\bar{X}$ ± S.D (°C)	Mean difference (°C)	*p*-value*
0.016	Opal	A_s	10	23.3 ± 0.9	17.7 ± 1.7	5.6	<0.001
		A_f	10	26.7 ± 0.5	27.6 ± 1.0	-0.9	0.022
	RMO	A_s	10	20.2 ± 1.2	17.6 ± 0.6	2.6	<0.001
		A_f	10	25.5 ± 1.3	25.5 ± 0.6	0.0	0.948
0.019 × 0.025	Opal	A_s	10	25.7 ± 1.1	15.7 ± 3.6	10.0	<0.001
		A_f	10	27.6 ± 0.9	28.6 ± 1.3	-1.0	0.050
	RMO	A_s	10	26.0 ± 1.6	17.3 ± 0.6	8.7	<0.001
		A_f	10	28.0 ± 1.8	25.9 ± 0.9	2.1	0.004

$\bar{X}$ ± S.D. stands for mean plus or minus standard deviation, and *N* is number of wires tested.
*Statistically significant at $p \leq 0.05$.

4.2. Comparison between transformation temperatures values using the BFR method

When comparing transformation temperatures of round and rectangular wires from the same manufacturer that were tested using the BFR method, statistically significant mean differences in the A_s values were seen between the round and rectangular wires for both Opal and RMO manufacturers. Likewise, the same was shown for the A_f values, as shown in Table 2.

Table 2. Comparison of transformation temperatures for BFR- tested round and rectangular wires of the same manufacturer.

Wire size comparison	Variable by manufacturer		Wire size (inches)	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*
0.016 versus 0.019 × 0.025	Opal	A_s	0.016	10	23.3 ± 0.9	-2.4	p ≤ 0.001
			0.019 × 0.025	10	25.7 ± 1.1		
		A_f	0.016	10	26.7 ± 0.5	-0.9	*p* = 0.024
			0.019 × 0.025	10	27.6 ± 0.9		
	RMO	A_s	0.016	10	20.2 ± 1.2	-5.8	*p* ≤ 0.001
			0.019 × 0.025	10	26.0 ± 1.6		
		A_f	0.016	10	25.3 ± 1.3	-2.5	*p* = 0.002
			0.019 × 0.025	10	28.0 ± 1.8		

$\bar{X}$ ± S.D. stands for mean plus or minus standard deviation, and N is number of wires tested.
*Statistically significant at $p \leq 0.05$.

When comparing transformation temperatures of rectangular wires from Opal with RMO using the BFR method, no statistically significant differences were found, as shown in Table 3; however, when making the same comparison with round wires, a statistically significant mean difference was seen for both A_s and A_f values between Opal and RMO manufacturers.

Table 3. Comparison of transformation temperatures for BFR-tested opal and RMO wires.

Manufacturers comparison	Variable by manufacturer		Manufacturer	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*
Opal vs RMO	0.016	A_s	Opal	10	23.3 ± 0.9	3.1	*p* ≤ 0.001
			RMO	10	20.2 ± 1.2		
		A_f	Opal	10	26.7 ± 0.5	1.2	*p* = 0.015
			RMO	10	25.5 ± 1.3		
	0.019 vs. 0.025	A_s	Opal	10	25.7 ± 1.1	-0.3	*p* = 0.663
			RMO	10	26.0 ± 1.6		
		A_f	Opal	10	27.6 ± 0.9	-0.4	*p* = 0.488
			RMO	10	28.0 ± 1.8		

$\bar{X}$ ± S.D. stands for mean plus or minus standard deviation, and *N* is number of wires tested.
*Statistically significant at $p \leq 0.05$.

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4.3. Comparison between transformation temperatures values using the DSC method

When comparing transformation temperatures of round and rectangular wires from the same manufacturer that were tested using the DSC method, no statistically significant mean differences in the A_s values were seen between the round and rectangular wires for both Opal and RMO manufacturers. Likewise, the same was shown for the A_f values, as shown in Table 4.

Table 4. Comparison of transformation temperatures for DSC-tested round and rectangular wires of the same manufacturer.

Wire size comparison	Variable by manufacturer	Wire size (inches)	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*
0.016 vs.0.019 × 0.025	Opal	A_s 0.016	10	17.7 ± 1.7	2.0	$p = 0.128$
		0.019 × 0.025	10	15.7 ± 3.6		
		A_f 0.016	10	27.6 ± 1.0	-1.0	$p = 0.058$
		0.019 × 0.025	10	28.6 ± 1.3		
	RMO	A_s 0.016	10	17.6 ± 0.6	0.3	$p = 0.281$
		0.019 × 0.025	10	17.3 ± 0.6		
		A_f 0.016	10	25.5 ± 0.6	-0.4	$p = 0.290$
		0.019 × 0.025	10	25.9 ± 0.9		

$\bar{X}$ ± S.D. stands for mean plus or minus standard deviation, and *N* is number of wires tested.
*Statistically significant at $p \leq 0.05$.

Also, when comparing transformation temperatures of round wires from Opal (17.7 °C) with RMO (17.6 °C) using the DSC method, no statistically significant difference was found between the A_s values. However, there was a statistically significant mean difference ($p \leq 0.001$) between the A_f values, with the Opal temperature being higher, as shown in Table 5. Likewise, when making the same comparison with rectangular wires, the same trend was observed. That is, the A_s values were not significantly different, but the A_f values were, with the Opal temperature being higher.

Table 5. Comparison of transformation temperatures for DSC-tested opal and RMO wires.

Manufacturer comparison	Variable by manufacturer	Manufacturer	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*
Opal vs RMO	0.016	A_s Opal	10	17.7 ± 1.7	0.1	p = 0.933

Manufacturer comparison	Variable by manufacturer	Manufacturer	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*
		RMO	10	17.6 ± 0.6		
		A_f Opal	10	27.6 ± 1.0	2.1	p ≤ 0.001
		RMO	10	25.5 ± 0.6		
	0.019 × 0.025	A_s Opal	10	15.7 ± 3.6	-1.7	p = 0.172
		RMO	10	17.3 ± 0.6		
		A_f Opal	10	28.6 ± 1.3	2.7	p ≤ 0.001
		RMO	10	25.9 ± 0.9		

$\bar{X}$ ± S.D. stands for mean plus or minus standard deviation, and *N* is number of wires tested.
*Statistically significant at $p \leq 0.05$.

4.4. Comparison between listed and tested transformation temperatures

Statistically significant mean differences were seen between Opal's listed A_s (20 °C) and A_f (37 °C) values[14] and the values obtained with BFR and DSC testing, as shown in Table 6. Furthermore, statistically significant mean differences were seen between RMO's listed A_f (32 °C) values[15] and the values obtained with BFR and DSC testing. No A_s values for RMO were listed by the manufacturer.

Table 6. Comparison between test method and manufactured listed transformation temperature.

Test	Variable by manufacturer	Listed mean value[a; b] (°C)	Wire size (inches)	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*	95% C.I.
BFR	Opal	A_s 20.0	0.016	10	23.3 ± 0.9	3.3	≤ 0.001	(2.7, 4.0)
			0.019 × 0.025	10	25.7 ± 1.1	5.7	≤ 0.001	(4.9, 6.5)
		A_f 37.0	0.016	10	26.7 ± 0.5	-10.3	≤ 0.001	(-10.6, -9.9)
			0.019 × 0.025	10	27.6 ± 0.9	-9.4	≤ 0.001	(-10.1, -8.8)
	RMO	A_f 32.0	0.016	10	25.5± 1.3	-6.5	≤ 0.001	(-7.4, -5.6)
			0.019 × 0.025	10	28.0 ± 1.8	-4.0	≤ 0.001	(-5.3, -2.7)
DSC	Opal	A_s 20.0	0.016	10	17.7 ± 1.7	-2.3	0.002	(-3.6, -1.1)
			0.019 × 0.025	10	15.7 ± 3.6	-4.3	0.004	(-6.9, -1.8)
		A_f 37.0	0.016	10	27.6 ± 1.0	-9.4	≤ 0.001	(-10.1, -8.7)

Test	Variable by manufacturer	Listed mean value[a; b] (°C)	Wire size (inches)	*N*	$\bar{X}$ ± S.D. (°C)	Mean difference (°C)	*p*-value*	95% C.I.
			0.019 × 0.025	10	28.6 ± 1.3	-8.4	≤ 0.001	(-9.3, -7.5)
	RMO	A_f 32.0	0.016	10	25.5 ± 0.6	-6.5	≤ 0.001	(-6.9, -6.0)
			0.019 × 0.025	10	25.9 ± 0.9	-6.1	≤ 0.001	(-6.7, -5.5)

$\bar{X}$ ± S.D. stands for mean plus or minus standard deviation, and *N* is number of wires tested.
*Statistically significant at $p \leq 0.05$.
[a]Opal Orthodontics by Ultradent Products, USA [Internet]. Heat Activated Nickel Titanium Arch Wires. c. 2015 [cited 2015 May 11]. Available from: http://www.opalorthodontics.com/products/arch-wires/via-wires/heat-activated-niti/Pages/default.aspx
[b]Laub, L.: Understanding titanium wires. The Orthodontic Cyber Journal. August, 2010. Ortho Cyber Journal, Inc. 6 January. 2013. <http://orthocj.com/2010/08/understanding-titanium-wires/> RMO did not provide any A_s values.

5. Discussion

In addition to an apparent lack of orthodontic literature using the bend and free recovery method to test heat-activated orthodontic archwires, there are few general studies that compare the BFR and DSC test methods. Therefore, a study comparing these two methods to test the transformation temperatures of as-received heat-activated archwires is of value.

Based on the literature, differences between the test methods were expected. However, studies that compare BFR and DSC show very similar results as long as the strain is no greater than 2.5%;[16;17;18] Butler et al., Chen et al., and Norwich have all compared A_f temperatures obtained from BFR and DSC testing.[16;17;18] Butler et al.[17] reported A_s values obtained using the BFR method, but stated that A_s could not be determined with DSC testing due to the presence of a rhombohedral phase (R phase) on the thermograms. Norwich did not provide transformation temperature values, but stated that the results "from each method agreed within one degree", while the other two studies' reported A_f temperatures showed differences ranging from 4 to 6 °C between the test methods.[16;17;18] Regardless of the differences, all three studies concluded that the values obtained by the two different methods corresponded with each other or were comparable.

Between the Butler et al., Chen et al., and Norwich studies, only the Chen et al. study reported a standard deviation; however, only the standard deviation for the BFR test method was reported (standard deviation of A_f was equal to 1.2 °C).[16;17;18] In this study, standard deviations for both methods are reported. Both BFR and DSC testing yielded relatively small standard deviations. For four of the eight test groups in this study, the BFR method resulted in lower standard deviations than the DSC method. Overall, the standard deviations averaged 1.2 °C for BFR testing and 1.3 °C for DSC testing. Such small standard deviations agree with the findings in the studies by Chen et al., Drexel et al., and the precision and bias statements in ASTM F 2004-05 and ASTM F 2082-06.[9;11;16;19] It is important to point out that the results in this study are reported to the precision of the thermocouple measurements for the BFR test method. Since it is the purpose of this study to investigate the suitability of the BFR method as a standard test method to determine the transformation temperatures of heat-activated NiTi orthodontic archwires, it is appropriate to report the transformation temperature values to the precision of the equipment used by the method, so they can be compared to values reported in other standard test methods, such as those in the precision and bias statements of ASTM F 2004 and ASTM F 2082, and the literature.[9;11;16;19] However, for clinical relevance, reporting transformation temperature values to the nearest degree Celsius is acceptable. For example, in the "Report" section of both ASTM F 2004-05 and ASTM F 2082-06, it is stated that "Temperature results should be reported to the nearest 1 °C" and "Results of the transformation measurements, reported to the nearest 1 °C", respectively.[9;11]

In this study, statistically significant differences were seen between the transformation temperatures obtained from the BFR and DSC test methods. However, the A_f temperatures obtained from the two methods were remarkably similar with the mean differences ranging from only 0.0 °C at the low end to 2.1 °C at the high end. As stated in ASTM F 2082 and ASTM F 2004,[9;11] the differences between transformation temperatures obtained with both test methods may be attributed to the effects of strain induced by BFR testing and possible cold work caused by the cutting of the specimen in DSC testing, respectively. Statistically significant mean differences between BFR and DSC testing were only seen for A_f values of round Opal wires and

rectangular RMO and Opal wires. However, according to the A_f results from both BFR and DSC testing, all of the tested wires will be fully austenitic below the average intraoral temperature range of 33−37 °C.[20]

While the A_f values obtained from BFR and DSC testing were clinically comparable, all of the A_s temperatures recorded from the two methods were significantly different. The variation between A_s temperatures between the two methods may have resulted from interpretation of the data on the DSC graph. The placement of the DSC tangent lines used to determine the A_s temperature was subject to interpretation between different specimens, since many of the plots showed double peaks due to the presence of an R-phase. Two-phase transformations make the determination of A_s more difficult. For example, when reporting transformation temperature values, Butler et al. did not report A_s values from DSC testing stating that, "The exact temperature, at which the austenite phase first occurs (A_s) is also unclear…due to the presence of an overlap between the completion of the rhombohedral phase and the onset of the austenitic phase."[17]

When comparing the transformation temperatures of round and rectangular wires from the same manufacturer, no statistically significant mean differences were seen when using the DSC test method (Table 4). However, when using the BFR test method to make the same comparison, statistically significant mean differences were observed between the transformation temperatures of round and rectangular wires from the same manufacturer in both instances (Table 2). In this study, since the same forming mandrel was used for all testing, the rectangular wires were subjected to slightly more strain, approximately 2.95% strain in comparison to the 2.5% strain of the round wires. Ruggedness testing for ASTM F 2082 showed that deformation strains above 2.5% resulted in a significant effect on A_s and A_f transformation temperatures.[10] This may explain why all of the transformation temperatures were higher for the rectangular (0.019in. × 0.025in.) wires in comparison to the round (0.016in.) wires when they were tested using the BFR method. Clinically, this means that deflecting orthodontic archwires of different sizes the same amount may affect the transformation temperature of one wire while having no effect on another, and it argues for the clinical relevance of the BFR test method. This phenomenon was demonstrated in another

study that showed an increase in the deformation strain from 2.4 to 5.8% increased A_f by approximately 1 °C independent of deformation temperature.[19] In the current study, although the increase in strain levels was small, approximately 0.5%, the measureable shift in BFR-tested transformation temperatures to higher values when comparing round to rectangular wires of the same manufacturer cannot be discounted. One significant advantage of the BFR method over the DSC method is its potential to be able to detect shifts in transformation temperatures of finished orthodontic archwires that are strained at different levels. For instance, if it is expected that a wire will be strained to high levels clinically (e.g., 5–6% instead of below 2.5%), then the finished orthodontic archwire can be tested at that strain level to get clinically relevant transformation temperatures that provide the clinician more accurate information about how the wire may behave in a patient's mouth.

Significant differences were observed between the manufacturer-listed transformation temperatures and those obtained with BFR and DSC testing for both manufacturers, as shown in Table 6. It can be seen that both the BFR and DSC tested A_f values were well below the listed values for both manufacturers. However, this is not true of the A_s values. RMO did not provide A_s values, which is typical for most manufacturers, and Opal lists its A_s value at 20 °C for both its round and rectangular wires.[14] From Table 6, it can be seen that the A_s values obtained from BFR testing are significantly higher than what Opal lists for its round and rectangular wires. On the other hand, DSC testing yielded significantly lower A_s values than what Opal lists for its round and rectangular wires. This temperature difference may be significant to the clinician. For example, for the rectangular Opal wire, the mean A_s value is 26 °C from the BFR method and 16 °C from the DSC method, while the manufacturer listed values are 20 °C.[14] For the scenario of a clinician's office temperature being at 20 °C, the DSC method predicts that the wire will have started to transition from the martensitic phase to the austenitic phase, while the BFR method predicts that the wire will still be in the martensitic phase.

Berzins and Roberts have previously tested the effects of thermocycling on the transformation temperatures of NiTi orthodontic wires.[21] However, this testing was done using DSC on small sections of the wires. An advantage of the bend and free recovery method that

was not investigated in the present study is that testing can be performed on wires that have actually been placed in patients' mouths. The intraoral environment subjects the wires to different pH levels, strains, and temperature fluctuations, which could affect the transformation temperatures of heat-activated NiTi orthodontic archwires. Using the recovery temperature testing apparatus, orthodontic archwires that have been placed in patients' mouths for different time intervals can be tested to examine if these wires are still active after an extended period of intraoral use.

6. Conclusions

This study showed that the bend and free recovery test method is a simple and practical technique that can be employed to measure the transformation temperatures of heat-activated NiTi orthodontic archwires. Overall, the average standard deviation for BFR testing was slightly lower than for DSC testing. Furthermore, A_f temperatures obtained from the BFR and DSC test methods were comparable with the mean differences ranging from 0.0 °C at the low end to 2.1 °C at the high end. Yet, the BFR method is a much more economical method.

There were, however, some notable positive differences between the BFR method and DSC method. A reported advantage of the BFR method over the DSC method is its potential ability to detect shifts in transformation temperatures of finished orthodontic archwires that are strained at different levels. This study did indeed show that deflecting heat-activated NiTi orthodontic archwires of different sizes the same amount, which results in a higher deformation strain for the larger wire, can raise the transformation temperature of the larger wire, which agrees with the literature and supports the clinical relevance of the BFR test method. Furthermore, this study showed that the BFR measured transformation temperature ranges for both archwire manufacturers were smaller than those measured by the DSC method. A reason for this is that the mean A_s values for both archwire manufacturers were significantly lower for the DSC method compared with the BFR method, which could effect the mechanical behavior of the archwires while the clinician is manipulating them. This is a topic for future research. To determine mechanical behavior of the archwires

at different temperatures, their load−displacement curves can be collected at various temperatures, including those below A_s, between A_s and A_f, and above A_f, as predicted by both the BFR and DSC methods.

The results of this study suggest that the bend and free recovery method is suitable as a standard method to evaluate the transformation temperatures of heat-activated NiTi orthodontic archwires.

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