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# Comparison of the transformation temperatures of heat-activated Nickel-Titanium orthodontic archwires by two different techniques

Noor Aminah Obaisi<sup>a</sup>, Maria Therese S. Galang-Boquiren<sup>a,\*</sup>,  
Carla A. Evans<sup>a</sup>, Tzong Guang Peter Tsay<sup>a</sup>, Grace Viana<sup>a</sup>,  
David Berzins<sup>b</sup>, Spiro Megremis<sup>c</sup>

<sup>a</sup> University of Illinois at Chicago Department of Orthodontics 801 S. Paulina St. Chicago, IL 60611, United States

<sup>b</sup> Marquette University 1250W. Wisconsin Ave. Wehr Physics Building 113A Milwaukee, WI 53233 United States

<sup>c</sup> American Dental Association 211 East Chicago Avenue Chicago, IL 60611-2678, United States

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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.

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\* Corresponding author at: University of Illinois at Chicago Department of Orthodontics 801 South Paulina Street Room 131 M/C 841 Chicago, IL 60611, United States. Tel.: +1 312 413 3022; fax: +1 312 996 0873.

E-mail address: [mgalang@uic.edu](mailto:mgalang@uic.edu) (M.T.S. Galang-Boquiren).

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## 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 is 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

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