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RESEARCH ARTICLE

Optimizing ageing conditions for commercial NiTi archwires: Insights from thermal phase transformation and tensile deformation analysis

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ABSTRACT - Superelastic nickel-titanium (NiTi) archwires are now commonly used as the standard archwire during the orthodontic alignment and levelling stage. They are preferred due to their ability to apply minimal force on teeth while allowing for a wide range of tooth movements. During orthodontic treatment, the orthodontist assesses the dimension and shape of the NiTi archwire to determine the amount and direction of force required to align misaligned teeth. The main contribution of this study is the parametric analysis and establishment of a set of optimal ageing temperatures and duration for the investigation of functionally graded nickel-titanium archwire using differential scanning calorimetry (DSC) and tensile deformation testing. The mechanical and thermal phase transformation behavior after ageing at six temperatures for duration of 15 minutes have been investigated using tensile deformation test and differential scanning calorimetry test in this paper. Experimental results reveal that in thermal analysis as the ageing temperatures increase from 400 °C to 490 °C, the austenite finish temperature rises to a value between 9.53 °C and 35.48 °C, and subsequently decreases to 520 °C. The archwire specimen aged for temperature of 490 °C exhibited the austenite finish temperature of around 35.48 °C, and it is highest among the aged wire specimens closest to oral temperature. In tensile deformation, the ideal ageing temperature for orthodontic applications was determined to be 490 °C for 15 minutes, resulting in relatively low plateau slope 13.73 GPa with high superelatic ratio 12.04, and maximum plateau strain of 7 %. This optimal ageing treatment ensures acceptable force delivery levels, maximum plateau strain, and minimal distortion in orthodontic treatment.

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1. INTRODUCTION

Nickel-Titanium shape memory alloys (SMAs) have found widespread application in orthodontics, orthopedics, and cardiovascular surgery due to their extraordinary shape memory behavior induced by martensitic transformation [1, 2]. These alloys are multi-functional materials exhibiting thermo-mechanical interaction which enables deformation recovery via unloading superelastic effect (SE) and heating shape memory effect (SME) [3]. During the initial levelling phase of orthodontic treatment nickel-titanium archwires are frequently employed due to their favorable mechanical properties which include shape memory, excellent spring-back and superelasticity, high elastic limit, and a low elastic modulus [4]. The temperature-induced crystallographic transformation from austenite to martensite is crucial because it gives rise to superelasticity [5]. Interestingly the maximum stresses that can be applied to nickel–titanium wires must be lower than the yield stress required to prevent dislocation slip in the martensite and austenite phases. Depending on the actual texture and strength, the maximum recoverable stresses of highly textured polycrystalline nickel–titanium wires resulting from the cubic to monoclinic martensitic transition reach between 8 % and 10 % [6].

Differential scanning calorimetry is the technique used to determine the crystalline phase of NiTi wires, as well as the transition temperature. Martensite start, M_s, martensite finish, M_f, austenite start, A_s and austenite finish, A_f are the phase transformation temperatures that can be examined using differential scanning calorimetry [7, 8]. Specifically, for orthodontic wires ANSI/ADA Standard No. 32 and ISO 1584 have outlined that DSC is the designated method for determining the austenite finish temperature, A_f for orthodontic archwires [9]. It's interesting that the key prerequisite NiTi alloy in orthodontics due to its superelastic characteristics at both room temperature and oral temperatures. Clinically it is important that when the A_f temperature of NiTi archwire closer to oral temperature then the archwire exhibits a martensite-austenite mixture making the wire malleable and easy to work with in crowded dentition [10].

The main focus of earlier research on the high strain rate deformation of shape SMAs was the mechanical behavior of these alloys at various strain rates and temperatures [8]. The ageing temperature has a significant impact on the precipitates' type, size, and distribution. The Ni_4Ti_3 precipitates with rhombohedral structure develop if the annealing temperature is in the range of 350 - 550 °C. If the annealing temperature is between 600 and 750 °C, Ni_4Ti_2 precipitates with a low-temperature orthorhombic structure and a high-temperature tetragonal structure will form. The Ni_3Ti

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precipitates occur with a hexagonal configuration if the ageing temperature is between 750 and 830 °C [11, 12]. Recent research has shown that extending the aging time duration causes the growth of Ni4Ti3 precipitates and a subsequent decrease in dislocation density which results in reduced superelasticity in NiTi alloys [13]. Therefore, by modifying the alloy composition and utilizing suitable aging treatment are effective methods for controlling microstructural parameters and achieving the desired material performance [14].

Recent research has focused on the effects of aging treatments on nickel-titanium shape memory alloys by impacting internal stress and chemical composition. Recent past investigation examined the elastic modulus of NiTi alloy by analyzing the initial slope which yielded a value of 35 GPa which was relatively low for the austenite phase [15]. It is well established that ageing limits shape memory alloys applicability to low-temperature applications due to their phase stabilization, transformation temperature hysteresis, and shape memory effect degradation at high temperatures. Ordering, martensite depletion, and austenite/martensite interface pinning are the fundamental effects of ageing treatment [16]. Gravina et al. [17] discovered, through a tensile test, that heat-activated NiTi archwires exhibited lower deactivation loadings compared to superelastic archwires. On the other hand, higher aging temperatures increased the plateau strain in stress-induced martensitic transition. Pseudoelastic deformation with a plateau strain over 12% occurred in the solution-treated sample, indicating plastic deformation [15, 18]. Controlled transformation temperatures up to 5.5% superelastic recovery were achievable with thermal treatments between 350 °C and 600 °C. It is worth mentioning that higher heat treatment temperatures have been found to reduce residual stresses in Ni-based super alloy by triggering the melt pool and dissolving dendritic structures [19].

To the best of the author's knowledge there have been no reports on the optimal heat treatment conditions for commercial NiTi archwires to achieve austenite finish temperature, A_f, low plateau slope and high superelastic ratio. The primary goal is to establish the optimal parameter, considering that heat-treatment temperature had a significant impact on thermo-mechanical properties in order to obtain adequate relatively low plateau slope, force delivery range and high superelastic ratio for orthodontic application. Therefore, before performing thermal and tensile deformation testing, the key target of the current study was to analyze Nickel–Titanium wires that had undergone a short-term ageing process at a broad range of temperature conditions. This experimental method is used to estimate the high super elasticity, low plateau slope, maximum unloading strain plateau, and A_f for a given ageing temperature and time by generating slope curves using universal testing machine (UTM) and differential scanning calorimetry.

2. MATERIALS AND METHODS

Experiments have incorporated the use of 0.4064 mm-diameter samples of commercial CE 0197 (ISO 13485) round natural superelastic upper nickel-titanium (NiTi) arch wire. Straight sections were cut from the as-received samples. Twenty arch wire samples were cut to approximately 50 mm in length and twenty samples of length (2-3 mm) were selected for DSC test including as- received wire and solution treated. The GSL-1100X furnace was used for solution treatment and ageing treatments at various temperatures in argon gas atmosphere. Selected straight samples were subjected to solution treatment at 920 °C for 20 minutes by adding titanium powder to a ceramic boat in furnace tube, followed by water quenching. The temperature range was 370 °C - 520 °C with temperature intervals of 30 °C, and all samples underwent ageing for 15 minutes. The schematic diagram of the present research work is depicted in Figure 1.

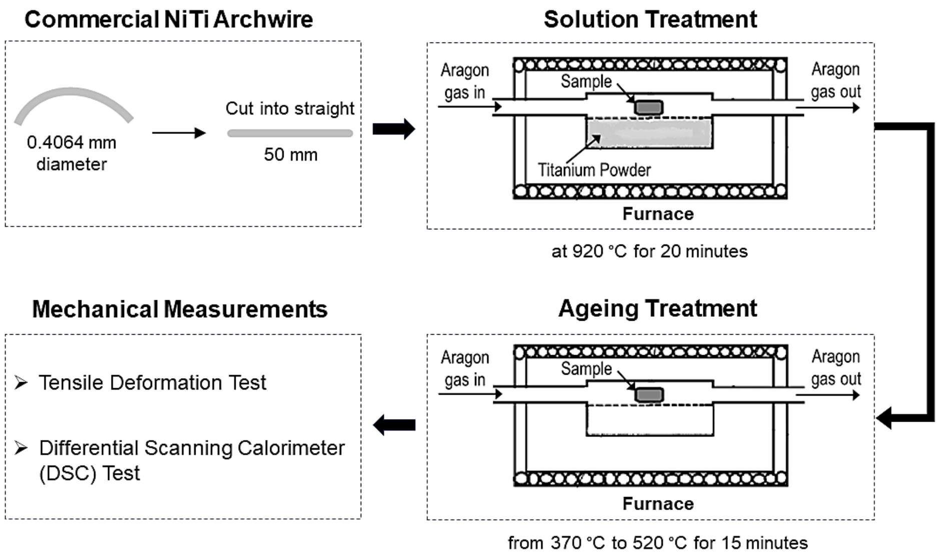

Figure 1. Schematic diagram of the present research work

The ISO-6892 compliant test sets for tensile deformation were conducted on the aged specimens using an Instron 3367 universal testing machine to determine their deformation behavior by setting elongation to 1.6 mm and the indenter and load cell to zero deflection in Bluehill v2.0 shown in Figure 2.

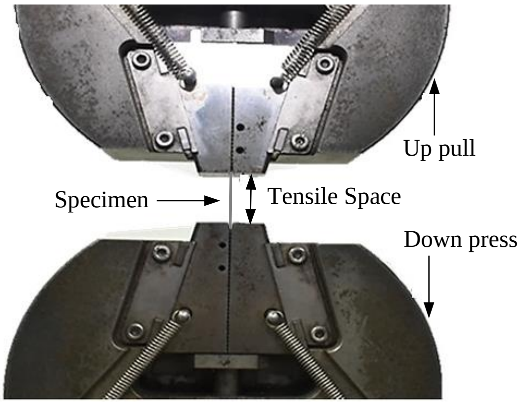

Figure 2. ISO standard 6892 NiTi archwire tensile deformation setup

In addition, thermal analysis was conducted to examine the phase transformation characteristics of as-received round NiTi archwire sample and aged samples. The TA Q20 differential scanning calorimeter was employed to determine the thermal phase transformation temperatures of both samples. These wire specimens were cut into lengths of approximately 2 to 3 mm, with masses ranging from 5 to 20 mg. The aged specimens' phase transition temperature was measured with a TA-Q20 differential scanning calorimeter at a heating and cooling rate of 10 °C per minute and a temperature range of -100 °C to 100 °C. A reference aluminum sample holder acted as an empty container in the DSC test cell while the wire segments for each test sample were placed in an aluminum sample holder. During the experiment, liquid nitrogen served as the coolant and nitrogen gas was used to purge the DSC cell at a rate of 50 mL/min. The fluctuations in temperature and enthalpy of phase transitions in NiTi wires were calculated using the computer program Universal Analysis. Every sample exhibited a thermograph, which was properly saved for further analysis. The start and finish temperatures of each phase transformation were determined using tangent lines and R phase indicates that NiTi alloy possesses the ability of two-way memory effect. The identification of peaks in the thermograms signified notable phase transformations within the aged archwires. The phase transformation temperatures of the NiTi alloy were precisely determined and quantified using the slope line method applied to these peaks [20].

3. RESULTS AND DISCUSSION

The present study was to evaluate the nickel–titanium wires that have undergone a thermal and tensile deformation test after being subjected to an ageing treatment at varying temperatures for short duration. The estimations of the transformation temperatures, i.e., M_s, M_f, A_s, and A_f, are based on the tangent line approach. The alloy demonstrates an austenite temperature finish and stress-strain curve of the alloy produced from an isothermal displacement (T = 298 K) controlled loading-unloading cycle up to a maximum deformation that coincides with the maximum deformation of the stress plateau in strain transformation. The subsequent sections detail the findings of tensile deformation and thermal transformation behaviour.

3.1 Tensile Deformation

The tensile tests were conducted in accordance with ISO specifications. The impact of temperature ageing on stress and strain during transformation, including reverse stress plateau and stress hysteresis. According to ISO standards, the stress hysteresis was defined in following expression:

$$SE = \frac{S_f}{S_p} \quad (1)$$

where, SE stands for superelastic ratio, S_f for the deactivation curve's final slope, and S_p for the plateau's slope. Using the best-fitting straight line with superimposition, a plateau phase was determined as the most horizontal region of a deactivation curve. The stress hysteresis, σ_h is determined by taking the difference between activation force, $F_{activation}$ and deactivation force, $F_{deactivation}$, as depicted in Eq. (2).

$$\sigma_h = F_{activation} - F_{deactivation} \quad (2)$$

The method utilized to calculate the transformation stress and strain values is in accordance with ASTM-F2516. According to Figure 3, the initial loading resulted in a forward plateau stress level of 3% strain, while the unloading curve resulted in a reverse plateau stress level of 2.5% strain. The plateau phase of deactivation curves was defined as the region of a deactivation curve that is the most horizontal and on which the best-fitting straight line is positioned. The results indicated that the condition of 490 °C /15 min produced the lowest plateau slope of all ageing temperatures, with a slope of 13.73 GPa and a SE ratio of 12.04, indicating "Superelasticity."

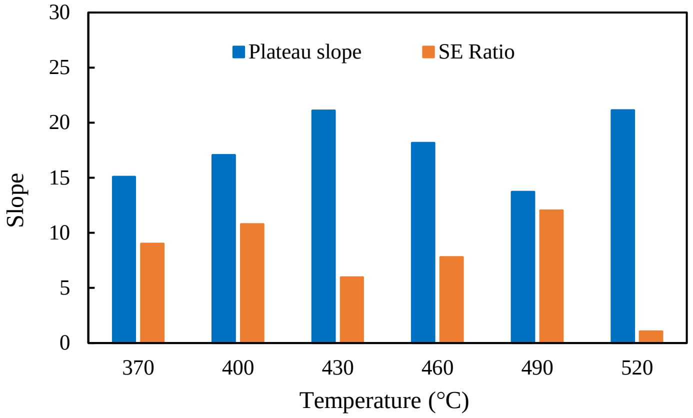

Figure 3. Plateau slope and SE ratio at different heat treatment condition

As a result, the specimens that experienced room-temperature deformation exhibited pseudoelastic behavior. Generally, the maximum forward plateau strain for specimens aged at 490 °C is 7%. However, as shown in Figure 4, specimens aged at 370 °C, 430 °C, and 520 °C showed a forward plateau strain of around 5%, while specimens aged at 400 °C and 460 °C had a plateau strain of 4%. Likewise, with the exception of the specimen aged at 490 °C, when stress increased the forward stress for transformation dropped correspondingly as the ageing temperature increased. At 520 °C, the highest level of residual strain was observed. This aligns with recent research findings which indicate that as the annealing temperature increases the residual strain becomes more prominent [21].

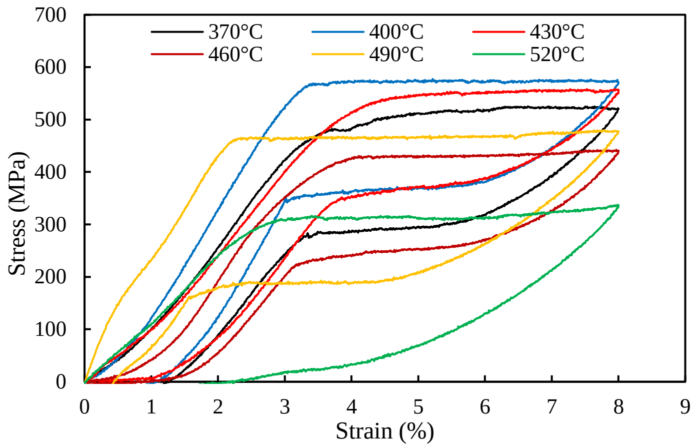

Figure 4. NiTi wire's stress-strain superelastic response at temperatures between 370 and 520 °C for 15 minutes

According to the ASTM specification [22] the upper plateau strength was defined as the stress at 3% strain during loading of the sample; and the lower plateau strength was defined as the stress at 2.5% strain during unloading after loading to 6% strain. The critical stress for forward and reverse transition corresponding as-received sample, as depicted in Figure 5. It has been noted that the transformation behavior in both phases of tensile stress significantly decreased when the ageing temperature increased from 370 °C to 430 °C and then gradually decreased to 460 °C to 520 °C. On the contrary, the reverse plateau slope's critical stress initially increases with an increase in ageing temperature up to 460 °C, and then decreases beyond it.

Additionally, the stress hysteresis observed in the pseudoelasticity demonstrated that the forward plateau strain and residual strain decreased as the aging temperature increased, up to 490°C, with a particularly significant peak residual strain at 520 °C. The results demonstrated that, among the specified temperatures, the 490 °C/15 min condition provided the lowest plateau slope. When NiTi wire is deformed under tension, it is generally believed that the inelastic stresses within the transformation/reorientation plateau range may be recovered upon unloading and heating. However, tensile deformation localized at the stress plateau need not be recoverable, especially if the NiTi wire is soft (tends to deform plastically at low stresses at room temperature) or if it is deformed at high stress/temperature [27]. In a recent study, it was found that the relationship between the superelastic nickel–titanium shape memory alloy's resistivity behavior and stress-induced martensitic transformation was investigated. Monitoring of strain-controlled low-cycle fatigue up to 6% [28]. Interestingly lower transformation strain could lead to lower pseudoelastic strain due to the narrow transformation range [29].

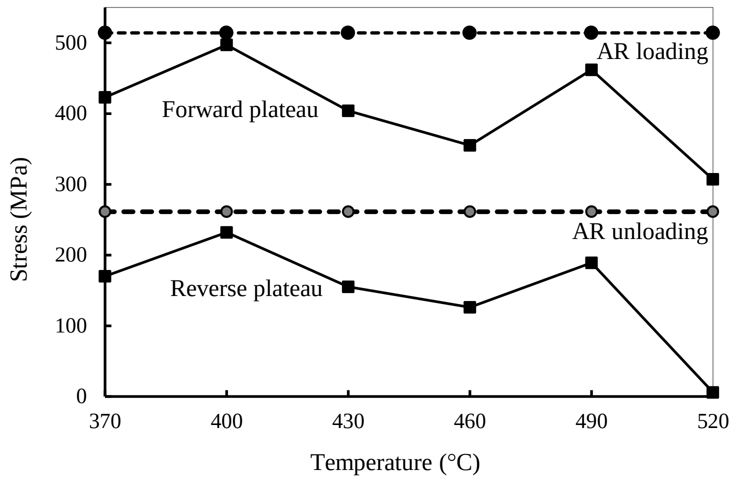

Figure 5. Effect of aging temperature on stress induced deformation for NiTi alloy aged for 15 minutes

The superelasticity of NiTi archwires is also assessed using the superelastic ratio for the unloading force. The SE ratio was determined in our study using the deactivation curves' final slopes since they seemed more reliable and consistent. More accuracy was also indicated by the ending slopes being more parallel and aligned to the onset of the activation curves. In this study, the SE ratio was calculated by dividing the deactivation curve's final slope by its plateau slope. The highest superelastic ratio was achieved by our commercial NiTi round wire at a heat-treatment temperature of 490° C.

3.2 Thermal Transformation Behavior

Round nickel-titanium orthodontic wire's thermal phase transformation behavior after 15 minutes of isothermal ageing at various temperatures. The as-received wire specimen exhibited two phase transformation peaks in the cooling and heating cycles, respectively. This indicates that the A↔M phase transformation occurs in two stages, while signifying the presence of an intermediate R-phase transformation. The A_f temperature of the as-received wire specimen was around 23.6 °C, which was close to and lower than the room temperature. Interestingly ageing at 460 °C and 490 °C resulted in multi peaks of transformation, which corresponds to the R-phase transition indicated in Figure 6.

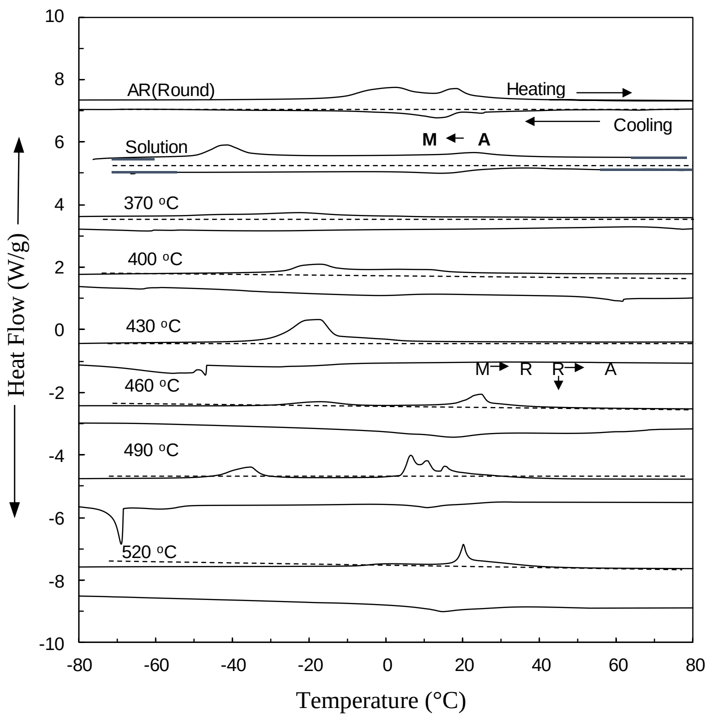

Figure 6. Thermal transformation behavior of the NiTi superelastic alloy after ageing for 15 minutes at various temperatures

Interestingly the ageing at temperature of 370 °C caused the significant reduction of A_f temperature to around -3.9 °C. Figure 7 illustrates that the A_f temperature rises to a value between 9.53 °C and 35.48 °C when the ageing temperatures rise from 400 °C to 490 °C successively, and then drops to 520 °C. The wire specimen that was aged for temperature of

490 °C exhibited the A_f temperature of around 35.48 °C, which was the highest among the aged wire specimens which is in accordance with previous research [23]. The identification of R-phase transformation in both the heating and cooling cycles of the aged wire was done via partial cooling and heating cycles, while confirming its persistence after aging treatment. It is worth mentioning that thermomechanical process has the potential to yield a material displaying superelastic behavior closely matching oral temperatures. This characteristic holds particular significance in dental applications, especially in the context of orthodontic archwires. The A_f temperature is crucial in both clinical and manufacturing settings. At this temperature, the alloy stabilizes and takes on its desired shape for the intended use [24].

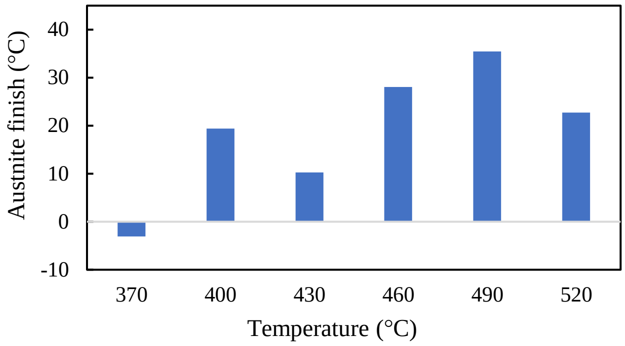

Figure 7. Thermal transformation behavior of the NiTi superelastic alloy after 15 minutes of ageing at various temperatures

Generally, 0.4064 mm nickel-titanium archwire since it is a commonly used archwire in clinical settings; consequently, the results may be extended to the majority of arches. In general, a difficult metallurgical process is involved in the production of nickel–titanium orthodontic archwires. The characteristics of the wires are influenced by their material composition, heat treatments, and thermomechanical procedures. In current study during thermal analysis, it has been observed that when the ageing temperatures increase gradually from 400 °C to 490 °C, the A_f temperature increases to 36 °C, which is close to body temperature, and then declines to 520 °C. The wire specimen that was aged at temperature of 490 °C had the highest A_f temperature of about 35.48 °C among the aged wire specimens. In order to produce adequate force delivery levels, a sufficiently low plateau slope, and a high super elasticity for orthodontic applications, the ideal temperature was determined to be 490 °C for 15 minutes. The lower load levels that relate to the A_f temperatures imply that the superelastic effect occurs at lower actuating force levels for the wire segments with higher A_f temperatures. It has recently been found that the archwire exhibited superelastic behavior at human body temperature. Because A_f is above room temperature, it is necessary to raise the temperature to generate the superelastic effect. At human body temperature each section is completely austenitic [26].

In addition, the findings reported above show that heat treatments affect the microstructures, phase transformation behaviours, and dynamic properties of nickel-titanium shape memory alloys. In Ni-rich nickel-titanium materials, it is well known that the mechanical characteristics are influenced by the grain size, dislocation distribution, and type, morphology, volume proportion, and size of precipitates. The critical stress for the stress-induced martensitic phase transition in ageing treated samples is higher than in homogenization-treated samples indicating that Ni_4Ti_3 precipitates are important in nickel-titanium during high strain rate deformation. According to existing research, when nickel-rich nickel-titanium alloys are subjected to quasi-static deformation, Ni_4Ti_3 precipitates dramatically change their mechanical properties [8]. Differential scanning calorimeter analysis was used to study phase changes in typical nickel-titanium wires. Endothermic peaks on the heating and cooling curves showed material structural changes. When nickel-titanium alloy is subjected to thermomechanical processing the phase composition can change the martensite or R-phase to manifest under clinical circumstances [25].

Therefore, the ageing temperatures of 490 °C demonstrated that a A_f temperature of around oral temperature is suitable for use in orthodontics. The A_f of NiTi alloys must be slightly below the oral temperature to enable superelastic behavior in orthodontic applications. As a result, for the NiTi archwires investigated in this study, heat treatment at 490 °C for 15 minutes would result in the best thermo-mechanical characteristics with the lowest plateau slope, the highest superelastic ratio, and an appropriate A_f for orthodontic tooth movement applications.

4. CONCLUSIONS

In this work, the pseudoelasticity of a nickel-titanium archwire that had undergone tensile deformation and thermal transformation was examined in relation to ageing time and temperature. In order to produce adequate force delivery levels, a relatively low plateau slope, and a high super elasticity as plateau slope 13.73 GPa and superelastic ratio 12.04, it was determined that 490 °C for 15 minutes was the optimum value. In thermal analysis, when the ageing temperatures increase sequentially from 400 °C to 490 °C, the A_f temperature rises to a value between 9.53 °C and 35.48 °C before decreasing to 520 °C. The wire specimen that was aged at a temperature of 490 °C had the highest A_f temperature of about 35.48 °C among the aged wire specimens. The main objective was to establish the ideal value, considering that heat-treatment temperature had a significant influence on thermo-mechanical characteristics, to offer acceptable force delivery levels, maximum plateau strain, and minimal distortion in orthodontic treatment. Therefore, among six ageing temperatures, 490 °C is chosen as the optimal temperature for orthodontic treatment.

5. ACKNOWLEDGMENTS

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