Force deflection comparison of various heat-activated superelastic nickel-titanium archwires: An *in-vitro* study

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Abstract

Objectives: To evaluate the load – deflections, compare superelasticity ratios and evaluate mechanical hysteresis for 5 commercial types of heat-activated superelastic orthodontic arch wires in a customized design of 3-point bend test maintained at a temperature of 37°C. Materials and Methods: The 3-point bending test was executed and provided values for the modulus of elasticity in bending, flexural stress, flexural strain and the flexural stress-strain response of the material. Results: Austenitic nickel-titanium (A-NiTi) exhibited constant forces but over a lesser range of deflection whereas heat-activated, superelastic nickel-titanium (NiTi) wires exhibited lower forces over a larger range of deflection. Conclusions: Heat-activated NiTi wires exhibited lower forces over a larger range of deflection, with the exception of one brand. In addition, they exhibit better superelastic ratios and a higher mechanical hysteresis than A-NiTi. A-NiTi exhibited constant forces but over a lesser range of deflection.

Key words: Load deflection, heat-activated, nickel-titanium, super elastic, hysteresis

INTRODUCTION

The constant unrelented search for an adept archwire, which can deliver optimal orthodontic force, has led to the invention of various multifunctional and innovative arrays of wires.

Orthodontic wire history starts from the use of piano wires, gold wires, stainless steel wires, [1] to the recently and widespread used nickel-titanium (NiTi) alloy, better known as Nitinol introduced by Dr. G. F. Andreasen in May 1971. Nitinol itself has undergone rapid alterations to be the supreme in clinical viability.

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A remarkable attribute of the Nitinol alloy is its "shapememory." This occurs when a material is deformed in its martensitic phase to allow better engagement of the wire for ligation and subsequently heated to its austenite finish (A_i) temperature after which the material then returns back to its original shape.^[1]

As is known, martensitic transformation and martensite reorientation are not perfectly reversible physical processes, so there always occurs a hysteresis. When a wire is activated, it causes a rapid increase in the load initially, which soon turns into a constant load over a long range despite increasing activation. When the wire is deactivated, the load decreases initially and then remains constant over a long range. The difference of the loading and unloading curves indicates the mechanical hysteresis in the wire.

To overcome this limitation, wires which are superelastic and with a better "springback" i.e., increased resistance to permanent deformation, with a lesser degree of mechanical hysteresis and better shape memory were developed.^[2]

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A difference in force levels exerted by wires from the same package has been reported. [3] It was also observed that the force values at mouth temperature can differ by 600% for wires of the same nominal diameter made by different manufacturers. [4] In the second of a two-part article, a variation for the intra-batch wires in the unloading plateau up to 10% has been reported. [5]

In effect, much remains to be ascertained as to how effectively the heat-activated superelastic NiTi wires made commercially available achieve the desired load – deflection characteristics across the spectra of assessment parameters.

AIM

To evaluate the load – deflections, compare superelasticity ratios and evaluate mechanical hysteresis for 5 commercial types of heat-activated superelastic orthodontic arch wires in a customized design of 3-point bend test maintained at a temperature of 37°C.

MATERIALS AND METHODS

The sample consisted of 100 NiTi archwires, of which 80 samples were heat-activated NiTi alloy and 20 samples of austenitic nickel-titanium (A-NiTi) alloy, which were tested in the "as received" condition. The wire dimension of 0.016×0.022 " was kept constant [Table 1]. These wires were chosen because of their vast clinical usage in slot leveling with both 0.018×0.025 " and 0.022×0.028 " appliance prescriptions.

The 3-point bending test provides values for the modulus of elasticity in bending, flexural stress, flexural strain and the flexural stress-strain response of the material. A custom

made device or bracket mount model was used to secure the archwires during testing. The jig assembly consisted of an upper member which was "L" shaped with an outer diameter of 3 mm. The upper end was milled with dimensions of $3 \text{ mm} \times 3 \text{ mm}$ for ease of clamping.

The lower member of the assembly was shaped to adhere 2 brackets in such a way, that the distance between the point of application and the supporting bracket was 7 mm. Two 0.018 × 0.025" standard-pre adjusted edgewise stainless steel twin brackets were bonded on either sides of the slot of the lower member with a cyanoacrylate adhesive (Fevikwik, Pidilite Industries Limited, India). The distance between the brackets was adjusted in such a way that the midaxes of the two brackets were 14 mm apart. Two brackets were oriented for flatwise testing [Figure 1].

The lower member was then attached to the stationary lower clamp and the upper member was attached to the load cell, with a full-scale range of 0-10 kg, of the cross head



Figure 1: Jigs with wire in place - start of loading

Group	Wire dimension	Branded as	Commercial manufacturer batch and lot details	Austenitic finish temperature (A,)
1	0.016×0.022"	Algn SE 200™	SDS Ormco Batch – 227-3136 Lot # – 00K143K	N/A
2	0.016×0.022"	Nickel-titanium memory wire heat-activated	American Orthodontics Batch – 857-942 Lot # – 9101	37°C
3	0.016×0.022"	Thermal activated nickel- titanium	Morelli Orthodontia Batch – 50.62.024 Lot # – 450781	35°C
4	0.016×0.022"	CopperNI-TI®	SDS Ormco Batch – 210-0932 Lot # – 05H225H	35°C
5	0.016×0.022"	Nitinol™ heat-activated	Heat-activated Nitinol 3M Unitek Batch – 4297-913 Lot # – AF6KG	32°C

of the Universal Testing Machine (Star Testing Systems No. STS – 248, Patiwana Group, India).

The "L" shaped hook of the upper member was positioned to pass freely through the groove of the lower member. The level of the upper member was adjusted to orient the hook midway between, and just below the level of the two brackets. A straight buccal segment of each wire was ligated in the brackets with a stainless steel 0.009" ligature. Each buccal segment of wire was oriented for flatwise loading.

On activation of the Universal testing machine, the "L" shaped hook was directed upward, pulling the wire along with it at a speed of 5 mm/min. The wire was deflected to a total of 5 mm from where the deflection reversed until the wire was unloaded to 0 mm.

The forces of loading and unloading were recorded by a software (STARTEST tensile software program, 1.0, Star Testing Systems 274/12, Vrindavan, Near Gokul Hall, Sion (E), Mumbai - 400 022, India), which directly procures information from the load cell of the machine at every 0.5

mm interval. These loading and unloading forces were plotted on the XY coordinate system. After each run, the ligation was released and the untested wires were subsequently mounted until all the samples were tested [Figures 2 and 3].

Since the temperature had to be maintained above the A_f temperature of the thermodynamic copper NiTi wires, the testing was carried out at 37°C in a temperature regulated chamber (No. 309 with a range of -10°C to 100°C) as an additional part of the apparatus during testing.

RESULTS

Results were computed and each group was compared with the control group, i.e., Group 1. Mean and standard deviation of loading and unloading forces, the superelasticity ratios and the mechanical hysteresis for each group was calculated and is reported in Tables 2 and 3.

An interesting phenomenon occurred during the deactivation of the 5 mm deflection for all wires tested. As the deactivation curve reached the 1.5-2 mm mark it showed a raise in force

Table 2: Means and SD of loading forces from Groups 1 to 5 at an interval of 0.5 mm from 0 to 5 mm deflection

Sample	e Group 1		Gro	Group 2		Group 3		Group 4		Group 5	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
0.5	170	38.93	171	30.07	202	42.74	171.5	47.38	171.5	69.60	0.019*
1.0	345.5	42.36	389	44.35	387.5	46.32	394	52.75	384.5	74.16	0.000*
1.5	427.5	36.68	530.5	41.10	494.5	67.78	515.5	38.86	534	66.60	0.000*
2.0	487.5	34.77	587	46.91	540	63.90	576	48.81	589	73.04	0.000*
2.5	512.5	39.31	614.5	48.50	557.5	58.83	615.5	58.17	634	70.44	0.000*
3.0	541	39.18	657	47.13	588	58.63	668.5	65.31	649	71.37	0.000*
3.5	577	43.05	700	51.91	604.5	56.79	705	71.19	684.5	66.05	0.000*
4.0	611	44.23	732	53.86	630	64.96	762	84.39	712.5	71.14	0.000*
4.5	644.5	47.95	786	49.88	652.5	62.73	815.5	109.47	741.5	82.92	0.000*
5.0	665	64.68	823.5	65.79	675.5	59.77	858	97.52	763	78.54	0.000*

 $^{{\}bf *Tested\ with\ ANOVA\ and\ Post\ Hoc\ Tukey\ comparison,\ Statistically\ significant\ at\ 95\ degrees\ of\ freedom,\ SD-Standard\ deviation$

Table 3: Means and SD of unloading forces from Groups 1 to 5 at an interval of 0.5 mm from 0 to 5 mm deflection

Sample	e Group 1		Gro	up 2	Gro	up 3	Gro	up 4	Gro	up 5	P value
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
5.0	665	64.68	823.5	65.79	675.5	59.77	858	97.52	763	78.54	0.000*
4.5	140.5	39.66	162	57.08	210.5	53.65	109	60.51	194	77.21	0.000*
4.0	102	36.36	113	48.46	165.5	43.10	102	46.85	158.5	69.30	0.000*
3.5	95	31.70	110.5	42.36	150.5	38.72	135	49.47	158	72.80	0.000*
3.0	107.5	35.37	116	42.84	156	51.74	181.5	43.31	167.5	77.38	0.000*
2.5	131	35.96	140.5	44.18	158.5	58.24	220.5	29.64	186.5	77.81	0.000*
2.0	147	28.11	159	47.78	173.5	52.24	249	41.15	208.5	73.93	0.000*
1.5	172	35.33	175.5	40.84	179.5	57.35	261.5	34.98	217	64.24	0.000*
1.0	155.5	29.81	143.5	29.06	166.5	41.07	215	39.66	150.5	57.71	0.004*
0.5	121	42.66	25.5	19.59	80.5	38.59	66.5	38.69	32	30.71	0.014*

^{*}Tested with ANOVA and Post Hoc Tukey comparison, Statistically significant at 95 degrees of freedom, SD – Standard deviation



Figure 2: Screenshot of computerized plotting of the load – deflection curve – start of loading

production. After 0.5-1 mm of deflection, the wires began to "recover" and produce average force levels of the plateau. Group 4 showed a steep raise in force production that ranged from 3.5 to 1 mm on unloading. This result is in agreement with the result obtained by a similar study. The reason attributed to this result is that forces generated depend on the severity of the deformation, the crystalline structure and the transition temperature range (TTR). It has been reported that the TTR could be shifted to a higher temperature in an alloy that forms stress induced martensite (SIM) during its activation. This would thus require a higher temperature than expected to transform the SIM into its austenitic form. In this study, the temperature was kept constant and above the A_r which explains the nature of the unloading curve.

Superelasticity ratio

The superelasticity ratio was computed as the ratio of the initial slope, which was marked on the graph as the point after which the graph showed a steep decline in the unloading curve and the final slope, which was marked on the graph as the point at which the load deflection was constant. It was noted that Group 1 exhibited a lower superelastic ratio than Groups 2, 3, 4, and 5. Among the thermal activated superelastic groups, Group 5 showed the least superelastic ratio of 4.8 as against a ratio of 4.3 for Group 1 (A-NiTi). The superelastic ratios for Groups 2, 3, and 4 were 5.6, 5.4, and 5, respectively [Table 4]. As is known, a material could be termed "superelastic" only if the ratio was above 8 and it was said to have superelastic tendencies if the ratio was above 2.^[8]

Mechanical hysteresis

The values of Mechanical Hysteresis were analyzed over a span of 0-5 mm at an interval of 0.5 mm. Since, the heat-activated NiTi wires exhibited higher loading forces than A-NiTi, the mechanical hysteresis of Groups 2, 3, 4, and 5 were greater than Group 1. This is not of significance as long as the unloading forces are light and constant.

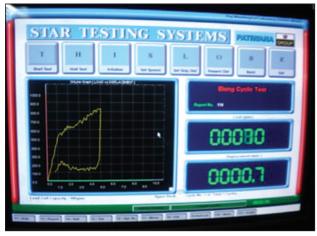


Figure 3: Screenshot of computerized plotting of the load – deflection curve – unloading

The value of maximum hysteresis for A-NiTi wires (Group 1) was 509 g at 4.5 mm deflection. The maximum hysteresis amongst the heat-activated NiTi wires (Groups 2, 3, 4 and 5) in the descending order were 454 g for Group 3 at 3.5 mm deflection, 547.5 g for Group 5 at 4.5 mm deflection, 624 g for Group 2 at 4.5 mm deflection, and 706.5 g for Group 4 at 4.5 mm deflection [Table 5]. The *P* value of all the values of mechanical hysteresis was found to be statistically significant at all deflections.

DISCUSSION

The Orthodontist's quest for developing an ideal aligning archwire included the property of delivering a constant force over a larger deflection and to resist permanent deformation; that is the material was expected to have a high springback. This came at the cost of formability in the form of copper added to NiTi. This wire has been manufactured with 4 different A_f temperatures: 15°C, 27°C, 35°C, and 40°C. The 35°C has been advised in most clinical situations. It has also been reported that in order to exploit the properties of this wire to its full potential, the working temperature of the appliance should be greater than the A_f temperature, since it is the difference between the oral temperature and the A temperature that determines the force generated by this alloy. To obtain reliable data, factors such as interbracket distance, type of bracket used, length of wire specimen, and method of ligation must be consistent. A 3-point bending test is a partially restrained model, has its benefits and drawbacks. The main advantage of a 3-point bend test is the ease of the specimen preparation and testing; however, the drawbacks are that the results of the testing method are sensitive to specimen and loading geometry and strain rate.

On loading the NiTi alloy, the austenitic phase transforms to SIM phase, which appears as a horizontal plateau, and

Table 4: Corroboration of the extent of the superelastic plateau and the superelasticity ratio									
Group	Initial slope	Start of plateau in mm	Termination of plateau in mm	Level of plateau in grams	Plateau slope	Superelasticity ratio			
1	1049	4	3	102	242	4.3			
2	1323	4	3	113	236	5.6			
3	930	4	2.5	165.5	172	5.4			
4	1498	4.5	4	109	297	5.0			
5	1138	4	2.5	158.5	237	4.8			

Table 5: The mechanical hysteresis calculated from 0 to 5 mm at an interval of 0.5 mm

Deflection	Group 1	Group 2	Group 3	Group 4	Group 5	P value
0.5	49	145.5	121.5	105	139.5	0.005*
1.0	190	245.5	221	179	234	0.000*
1.5	255.5	355	315	254	317	0.000*
2.0	340.5	428	366.5	327	380.5	0.000*
2.5	381.5	474	399	395	447.5	0.000*
3.0	433.5	541	432	487	481.5	0.000*
3.5	482	589.5	454	570	526.5	0.000*
4.0	509	619	464.5	660	554	0.000*
4.5	504	624	442	706.5	547.5	0.000*
5.0	0	0	0	0	0	0.000*

*Tested with ANOVA and Post Hoc Tukey comparison, Statistically significant at 95 degrees of freedom

this is the indication of the expression of superelastic properties. Each wire has a specific TTR in which the phase transition takes place.

Superelastic compounds generally present a high stiffness in the initial segment of the slope of the stress-strain graph when the deflection of the wire is still minimum. The initial activation force required for A-NiTi could be 3 times greater than the force required to deflect a work hardened martensitic wire. However, when the SIM is formed, the horizontal plateau appears and the alloy "absorbs" any additional load stress and releases it in constant amounts during the deactivation phase. Actually, the linear region corresponding to the deactivation plateau is lower than the activation plateau and is parallel to it. This phenomenon is called hysteresis. The main clinical interest of hysteresis is that the force delivered to the periodontal structures is lower than the force required to activate the wire. Furthermore, the unloading force is the force that is applied by the wire to the tooth to cause its movement. [9]

A study was conducted to determine the effect of ligation on the load – deflection characteristics of NiTi orthodontic wire using a 3-point bending system where Steel ligation was pitted against elastomeric modules and a slot lid. The results indicated that the steel ligation of brackets might result in the NiTi wire exhibiting significantly heavier loads and significantly different load-deflection curves from those traditionally expected. The load traditionally expected to be

generated by the true properties of the NiTi wire may be doubled or tripled by the act of ligation when measured in the unloading plateau region. The elastomeric module may act as a restraint on NiTi wire by limiting superelasticity. Hence, Steel ligation was preferred for this study.^[10]

To eliminate any bias toward a particular manufacturer, the operator was blinded from the wire, which was being tested. The wires were color coded before the test and were decoded after all the samples were tested.

It was unexpected to see that heat-activated NiTi wires exhibited higher forces than A-NiTi in all groups. This could have happened due to the contribution of steel ligation, which tends to increase the force exhibited by superelastic NiTi wires.^[10] This is not in corroboration with previous studies. It was only at a deflection of 0.5 mm on the unloading curve, that A-NiTi exhibited higher forces than the heat-activated NiTi wires. In NiTi wires, the final part of the deactivation curve, where the material is usually in austenitic phase, is almost linear and resembles that of stainless steel or conventional work hardened NiTi.[1] In a clinical situation, if the wire is engaged to mildly crowded teeth, the NiTi will behave like other materials. However, in superelastic NiTi, the final part of the unloading curve will be nonlinear exhibiting superelasticity and better leveling characteristics. Therefore, as expected in the unloading value of 0.5 mm, A-NiTi had the highest value than all the heat-activated NiTi groups. This is in agreement with the previous studies.[4,5,11]

The superelasticity ratio is a measure of the ratio of the final part of the deactivation curve to the slope of the plateau. A conventional material such as stainless steel will give a ratio of about 1, while an ideal superelastic material will give a ratio of infinity. By definition, a material is said to have superelastic tendencies when the ratio is above 2 and if the ratio is above 8 is said have superelasticity. It was observed that amongst all the other thermal activated superelastic groups, Group 5 exhibited the least superelastic ratio of 4.8 as against 5.6, 5.4, and 5 for Groups 2, 3, and 4, respectively. Group 1, which has an austenitic grain structure, exhibited a superelastic ratio of 4.3. This clearly discerns that thermal activated NiTi wires exhibit a higher superelastic ratio than A-NiTi. It was observed

that Group 4 had the termination of the unloading curve starting at a higher deflection than A-NiTi. This suggests the limited range over which this group can deliver light and continuous forces. Conversely, Groups 3 and 5 showed a longer range over which they could express light and continuous forces. As for, Group 2, it had a range similar to that of Group 1 (A-NiTi).

The start of the plateau indicates the highest deflection at which the wire will exhibit low and constant forces. ^[8] Unexpectedly, all the heat-activated NiTi groups exhibited the start of the plateau at the same deflections as A-NiTi. This means that in cases of severe crowding, the copper NiTi wires will exhibit light and continuous forces as A-NiTi when deflected to the same distance.

Groups 3 and 5 exhibited a higher force level than that of A-NiTi. This was completely unexpected. Two factors may explain this behavior. During the manufacturing process some parameters may not have been adjusted optimally, which would result in less than ideal product properties. Minute differences in the manufacturing process, have a significant impact on the behaviour of the product.^[4,5]

Superelastic alloys also exhibit hysteresis, that is the activation and deactivation plateaus have different stress magnitudes. As a result, the wire does not deliver the same force as that applied to activate it. Hysteresis can also be thought of as the friction associated with the movement of twin related martensite boundaries. The magnitude of hysteresis depends on the alloy composition.

Again, unexpectedly the heat-activated NiTi wires showed a higher mechanical hysteresis at all deflections as compared to A-NiTi. This result may be attributed to friction, which increased the force during loading and decreases the force during unloading.^[8] Clinically, this might not be significant.

One may wonder if such a rigid evaluation of the superelastic property of the alloys is truly necessary in clinical applications. The performance of any NiTi alloy is strictly influenced by its composition and manufacturing procedures. For example, in theory, it is possible that one specific alloy in its austenitic phase (or even a work hardened alloy) could deliver a biologically acceptable force, comparable to the force of a superelastic alloy in phase transition, especially when only moderate deflection is necessary. Despite commonly accepted commercial claims, the low values of force delivery of most NiTi alloys still need to be precisely quantified and compared with the force delivery of other established alloys through properly designed experiments.

CONCLUSION

- 1. Heat-activated NiTi wires exhibited lower forces over a larger range of deflection, with the exception of Group 4.
- Groups 3 and 5 exhibited a higher force level during unloading as compared to Group 1. Clinically, this may not be desirable as it could have deleterious effects on the periodontium.
- 3. A-NiTi exhibited constant forces but over a lesser range of deflection. Clinically, this means that in a case of severe crowding, this wire may not exhibit a constant force over a large range of activation and may even exert a higher than desired force.
- 4. Heat-activated NiTi wires exhibited marginally better superelastic ratios than A-NiTi wire. Clinically, this implies that in a case of a severely crowded arch, the HANT wires will ensure a better engagement of the wire during ligation.
- 5. Heat-activated NiTi exhibited higher value of mechanical hysteresis as against A-NiTi wire. This is insignificant clinically.

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