Transformation Behaviour, Bending Properties and Surface Quality of 22 Commercial Nickel-Titanium Wires: A Batch-to-Batch Evaluation

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Authors’ contributions

This work was carried out in collaboration between all authors. Authors VB, IG, MV and PL contributed equally to this study. All authors read and approved the final manuscript.

ABSTRACT

Aims: The aims of this study were to investigate batch-to-batch differences in mechanical and thermal properties of 22 commercial nickel-titanium wires and evaluate the surface quality. Secondly the percentages of superelasticity at mouth temperature were measured.

Study Design: In vitro laboratory study.

Place and Duration of Study: Sample: Department of Oral Health Sciences – Orthodontics and Metallurgy and Materials Engineering KU Leuven, between January 2010 and May 2011.

Methodology: The sample comprised 22 NiTi wires of the upper jaw from 7 different manufacturers. For each brand two different batches were investigated (LOT A, LOT B). Three different investigations were carried out. First transformation behaviour and transition temperatures were determined by differential scanning calorimetry, second a three point bending test was done to evaluate the mechanical properties and third scanning electron microscopy was performed on all samples to evaluate surface quality.

Results: Absolute batch-to-batch differences in the investigated properties were all deviating from zero, although most differences were clinically acceptable. Significant interbatch differences in thermal properties were found in 4 wires. Mechanical interbatch differences were found remarkable for 5 wires. Percentage of elasticity showed a non-

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superelastic curve below a deflection of 1500 µm, which represents clinical small deflections. Surface topography showed also interbatch differences for 4 wires.

**Conclusion:** This study shows that batch-to-batch differences are obvious in each sample. One has to be careful in the selection of archwires. Therefore more standardized consumer information should be provided.

**Keywords:** Mechanical properties; thermal properties; nickel-titanium wire; three point bending; load deflection.

1. **INTRODUCTION**

Ideally an orthodontic force should accomplish a rapid tooth movement with minimum patient discomfort and without damage to the teeth and periodontium. Some evidence exists that this may be achieved with small and continuous forces (Daskalogianakis and McLachlan, 1996; Darendeliler et al., 1997; Owman-Mol, et al., 1995). The armamentarium of the orthodontist today consists mainly of four different archwire alloys. Those are nickel-titanium, stainless steel, cobalt-chromium and beta-titanium. Especially nickel-titanium wires have been of considerable interest to the specialty since the introduction by Andreasen and Hilleman (1971).

The first nickel-titanium wires were marketed by the Unitek Corporation with the name 'Nitinol' (NiTi Naval Ordnance Laboratory) (e.g. Nitinol SE, 3M Unitek, Monrovia, California, USA). Through a work hardening process these wires achieve three important characteristics for orthodontic use, namely a reasonable strength, low stiffness (its modulus of elasticity is about one sixth that of stainless steel) and a large springback allowing a large deflection without permanent deformation. Disadvantages are lack of both weldability and formability which is defined as the amount of permanent deformation that a wire can withstand before failing (Brantley, 2001).

By the 1980s however, two active nickel-titanium wires, were introduced: martensitic-active wires (e.g. Sentaloy, GAC, Central Islip, New York, USA) and austenitic-active wires (e.g. NiTi, Ormco, Glendora, California, USA). Each of these alloys exhibit two additional properties, unique in dentistry and beneficial for orthodontic treatment: the shape memory effect (SME) and superelasticity (SE) (Proffit, 2007).

These properties are attributed to a phase transformation between the body-centered cubic austenitic form, existing at higher temperatures, and the monoclinic martensitic form, existing at lower temperatures. This 'martensitic transformation' is influenced by changes in temperature and stress. 'Nitinol', on the other hand being cold formed, was marketed in a stabilized martensitic crystal structure, being not transformable. In addition to martensitic and austenitic phases, an intermediate phase with rhombohedral symmetry has been described as the R-phase (Miyazaki and Otsuka, 1989).

Some superelastic NiTi wires contain an amount of copper (5-6 %) to increase strength and to reduce energy loss. Unfortunately these benefits are associated with an increase in the phase Transformation Temperature Range (TTR) above that of the mouth temperature. Therefore 0,5% chromium is added to reduce its TTR (Parviziand Rock, 2003).

NiTi alloys belong to a larger group of shape memory alloys as they literally have the capacity to remember their original shape. In a typical application, a certain shape is set
while the alloy is maintained at an elevated temperature, above the martensite-austenite transition temperature. Moreover, this temperature is quite low (-80°C to 100°C) compared to the transition temperature of martensite in steel ranging from 200 to 800°C. It should be noted also that the martensite in steel is of a different kind as the martensite in NiTi. Steel does not exhibit any shape memory effect or superelasticity. When the alloy is cooled below the transition temperature it is quite soft and can be apparently plastically deformed by reorientation of the martensite variants, making it easier to ligate the archwire in the brackets. In the mouth, where the wire is heated again by body temperature above its transition temperature, the original shape is restored aligning the teeth at moderate forces (Honma, 1979; Wayman, 1980; Saburi, 1985).

In orthodontics this thermoelastic property is especially expressed in a specific type of NiTi-wire, having a martensite active phase at room temperature: the heat-activated (HA) NiTi wires (e.g. Sentaloy, GAC, Central Islip, New York, USA).

The second beneficial property of NiTi wires having an austenitic-active structure at room temperature (e.g. NiTi, Ormco, Glendora, California, USA) is their super- or pseudoelasticity at a broad temperature range.

Opposite to stainless steel alloys, NiTi wires when ligated into the brackets undergo a phase transformation from austenite to stress-induced martensite. As the teeth start to move and the stress is released, the reverse transformation to the parent phase causes an unloading plateau, where the force exercised by the NiTi wire hardly varies over a considerable range of deflection. Due to this high springback property (restoring potential), no permanent deformation occurs (Andreasen and Morrow, 1978). However, a survey of the properties of superelastic wires in vitro is necessary because some researchers concluded that few of these wires actually exhibit superelastic properties (Meling and Ødegaard, 1998; Tonner and Waters, 1994; Segner and Ibe, 1995; Schumacher et al., 1992). Those laboratory findings were supported by clinical studies in which no significant differences of tooth migration were observed among superelastic NiTi wires and comparable conventional NiTi wires (Brantley, 2001; Cobb et al., 1998; Evans et al., 1998).

Comparative studies have been carried out in the past evaluating the mechanical and thermal properties of different brands of NiTi orthodontic devices (Fischer-Brandies et al., 2003; Bourauel et al., 1997; Langeron, 1999). However, for NiTi alloys few batch-to-batch evaluations have been accomplished investigating the accuracy of the manufacturing processes (Bourauel et al., 1997).

The purpose of this study was twofold. First batch-to-batch comparison of various nickel-titanium wires were carried out by conducting differential scanning calorimetry (DSC), three point bending tests (DMA) and scanning electron microscopy (SEM). Second, each wire was investigated for its percentage of superelastic effect at a minimum standard deflection, measured at mouth temperature (37°C).

**2. MATERIALS AND METHODS**

In total 22 commercially available superelastic, heat-activated and martensite stabilized NiTi archwires of the upper jaw from 7 different manufacturers were selected for this investigation (Table 1, Table 2). From each brand two different batches were examined (LOT A and B).
Table 1. Different manufactures

| Supplier                      | City        | State     | Country |
|-------------------------------|-------------|-----------|---------|
| 3M Unitek                    | Monrovia    | California| USA     |
| Leone                         | Firenze     | Italy     |         |
| G&H Wire Company             | Franklin    | Indiana   | USA     |
| Ormco                        | Glendora    | California| USA     |
| Highland Metals Inc          | San Jose    | California| USA     |
| Dentaurum                    | Pforzheim   | Germany   |         |
| Ortho Organizers             | Carlsbad    | USA       |         |

Table 2. 22 different wires. For each wire supplier, LOT numbers and properties were summarized. (HA= heat-activated, SE= superelastic, Cu= Copper)

| Wire          | Supplier                      | Brand          | HA  | SE  | Cu  | ref nr  | LOT A     | LOT B     |
|---------------|-------------------------------|----------------|-----|-----|-----|---------|-----------|-----------|
| Rectangular wires: 0.016 x 0.022 inch |
| 1             | 3M                            | Nitinol        | HA  |     |     | 4297-913| L7598     | M6405     |
| 2             | 3M                            | Nitinol CLASSIC|     |     |     | 297-171 | Z4891     | A0013     |
| 3             | Leone                         | Memoria        |     |     |     | C5912-16| 05062801 | 05102001  |
| 4             | G&H Wire Company              | Thermal NiTi   | HA  |     |     | TEFU1622| 76213     | 75167     |
| 5             | G&H Wire Company              | NiTi           |     | SE  |     | SSEEFU1622| 75818     | 77000     |
| 6             | G&H Wire Company              | Triple Force NiTi| SE |     |     | TTFU1622| 161944    | 158197    |
| 7             | Ormco                         | Co NiTi 40ºC   | Cu  |     |     | 210-0942| 04M194M   | 05DS91D   |
| 8             | Ormco                         | Co NiTi 35ºC   | Cu  |     |     | 210-0922| 05C45     | 05B67     |
| 9             | Ormco                         | Co NiTi 27ºC   | Cu  |     |     | 210-0902| 05C128C   | 04M384M   |
| 10            | Ormco                         | NiTi           |     |     |     | 210-0502| 04L455L   | 05A560A   |
| 11            | Highland Metals Inc           | NiTi arches    | HA  |     |     | 17163    | 17164     |           |
| 12            | Dentaurum                     | Rematitan Lite | SE  |     |     | 766-090-01| 41033     | 45393     |
| 13            | Ortho Organizers              | Nitanium       |     |     |     | 100-662  | 377425c05| 388546A06 |
| Round wires: 0.016 inch |
| 14            | 3M                            | Nitinol CLASSIC|     |     |     | 296-161  | Z7339     | D5730     |
| 15            | 3M                            | Nitinol        |     | SE  |     | 4296-913| N5542     | R8566     |
| 16            | Leone                         | Memoria        |     |     |     | C5910-16| 04111001 | 05102001  |
| 17            | G&H Wire Company              | Thermal NiTi   | HA  |     |     | TEFU016 | 73820     | 78952     |
| 18            | G&H Wire Company              | NiTi           |     | SE  |     | SSEEFU016| 65336     | 80298     |
| 19            | Ormco                         | Co NiTi 35ºC   | Cu  |     |     | 205-0064| 05C45C    | 05A473A   |
| 20            | Ormco                         | NiTi           |     |     |     | 205-0001| 04M353M   | 05C180C   |
| 21            | Dentaurum                     | Rematitan Lite | SE  |     |     | 766-082-01| 413225    | 46591     |
| 22            | Ortho Organizers              | Nitanium       |     |     |     | 100-652  | 377675c05| 387259A06 |

The samples were divided in two groups: numbers 1 – 13 were rectangular wires (0.016 x 0.022 inch) and numbers 14 – 22 were round wires (0.016 inch). Occluso-gingival and bucco-lingual dimensions were measured using an Electronic Digital Calliper (Orteam, Milano, Italy) with an accuracy of 0.01 mm. Every wire was weighted and its weight was expressed in grams. Samples of each wire brand were submitted to three different investigations.
2.1 Differential Scanning Calorimetry

First for each sample the transformation behaviour and the transition temperatures were determined by thermal analysis. This was investigated with differential scanning calorimetry at MTM KU Leuven (2920 MDSC V2.6A TA instruments, New Castle, Delaware, USA) based on ASTM Standards F2004. The amount of heat released or absorbed during phase transformation (heat flow W/g) was recorded as a function of the temperature (°C) in a range from -90°C to +90°C. The transition temperature was recorded at the start (s) and at the finish (f) for both the austenitic (A), martensitic (M) and transitional (R) phase (As, Af, Ms, Mf, Rs, Rf). They were determined according to DIN 51007 using appropriate software. Two measurements per wire sample were made (one in the front and one in the end), but only proceeded until wire 11, since no significant differences were observed. Measurement runs were performed per sample, with a heating and cooling rate of 10°C/min under a dry nitrogen purge.

2.2 Three Point Bending Test

The mechanical bending properties of the samples were determined by a dynamic mechanical analyser (DMA, Q800 V7.0 Build 113, TA instruments, New Castle, Delaware, USA) in conjunction with a liquid nitrogen cooling system. The three point bending test has been proven to be a valid instrument, reproducing clinical environment conditions (Miura et al., 1986).

The rectangular wires (0.016 x 0.022 inch) were measured under vertical loads applied on their flat, wide sides (0.0022 inch). The samples were supported at both ends, having a beam length of 20 mm. The mid portion of the wire segment was deflected at a cross-head speed of 200 µm/min, with a marge of 5 µm, with a DMS load gauge of maximum 18 N from a metal pole of 5 mm in diameter. Each sample was deflected until 1500, 2500 and maximum 3500 micrometer was reached. The static plateau forces were measured.

The samples were unloaded at the same cross-head speed until the loading force became zero. The loading and unloading tests were performed at 22°C, 37°C and 60°C.

2.3 Scanning Electron Microscopy

Additionally scanning electron microscopy (SEM) was performed on all samples to evaluate surface quality with the intention of localizing remarkable manufacturing defects when present. Surface topography was analyzed using a Philips XL 30 FEG (Philips, Amsterdam, The Netherlands) SEM at magnifications of 2000 times.

As insufficient amounts of material was available (only for the DSC procedure different batches of wires 1 and 3-10 were tested twice), only descriptive statistics have been performed.
3. RESULTS

3.1 Differential Scanning Calorimetry

With the DSC testing procedure the transition temperature range (TTR) for each LOT was analysed (example: Fig. 1). In Table 3 the wires were summarized according to their transition properties.

![DSC graph](image)

**Sample:** wire 1, LOT B, measurement 4  
**Size:** 3.7000mg  
**Instrument:** 2920 MDSC V2.6A

Fig. 1. Heating and cooling diagram of wire 1, LOT B. Cooling and heating direction were marked with arrows. This wire shows 3 transition phases. After heating the wire will transform to an austenitic phase. This phase starts with an austenitic starting temperature (As) and ends with an austenitic finishing temperature (Af). From here on the wire is 100% austenite. When the wire is cooled, it will be transformed into the martensitic phase, again with starting temperature (Ms) and finishing temperature (Mf). This wire also has an R-phase transition with its Rs and Rf before it transformed to the martensitic phase, while it was cooling down.

The Nitinol classic (2, 14) and the Nitinol superelastic (15) are the only investigated archwires which didn’t show a transition in the temperature range under investigation (-90°C to +90°C). All the other archwires showed martensitic and austenitic transformations and some of them also a R-phase transition.
Table 3. Differential scanning calorimetry testing: summarized transition properties of the 22 tested wires. * Interbatch differences. The first six wires show only a martensitic phase (M) and an austenitic phase (A). Fifteen wires have also an R-phase transition (R) and three wires show no transition in the temperature range of this investigation.

| Wire | Without R-phase transition (normal martensitic-austenitic transformation) | With R-phase transition (one- or two-step transformation) | With NO transition in the temperature range under investigation (-90°C to +90°C) |
|------|--------------------------------------------------------------------------|-----------------------------------------------------------|--------------------------------------------------------------------------------|
| 7    | Co NiTi 40°C 0.016 x 0.022 inch Heating: M => A                          | NiTi 0.016 inch Heating: M => A => R => M                  | NiTi CLASSIC 0.016 x 0.022 inch                                                |
| 8    | Co NiTi 35°C 0.016 x 0.022 inch Heating: M => A                          | NiTi 0.016 inch Heating: M => A => R                       | NiTi CLASSIC 0.016 inch                                                        |
| 9    | Co NiTi 27°C 0.016 x 0.022 inch Heating: M => A                          | Memoria 0.016 inch Heating: M => A => R                    | NiTinol 0.016 inch                                                            |
| 19   | Co NiTi 35°C 0.016 inch Heating: M => A                                  | Thermal NiTi 0.016 inch Heating: M => A => R               | Nitinol 0.016 x 0.022 inch                                                     |
| 13   | Nitanium 0.016 x 0.022 inch Heating: M => A                             | Nitanium 0.016 inch Heating: M => A                         | Nitinol CLASSIC 0.016 inch                                                    |
| 22   | Nitanium 0.016 inch Heating: M => A (LOT A)*                             | Triple Force NiTi 0.016 inch Heating: M => A => R          | Nitinol 0.016 inch                                                            |
|      |                                                                          | 11 NiTi 0.016 x 0.022 inch Heating: M => A => R            | Nitinol 0.016 inch                                                            |
|      |                                                                          | 12 Rematitan Lite 0.016 inch Heating: M => A => R          | Nitinol CLASSIC 0.016 inch                                                    |
|      |                                                                          | 16 Memoria 0.016 inch Heating: M => A => R                 | Nitinol 0.016 inch                                                            |
|      |                                                                          | 17 Thermal NiTi 0.016 inch Heating: M => A => R            | Nitinol CLASSIC 0.016 inch                                                    |
|      |                                                                          | 18 NiTi 0.016 inch Heating: M => A => R                    | Nitinol 0.016 inch                                                            |
|      |                                                                          | 20 NiTi 0.016 inch Heating: M => A => R                    | Nitinol CLASSIC 0.016 inch                                                    |
|      |                                                                          | 22 Nitanium 0.016 inch Heating: M => A => R (LOT B)*      | Nitinol 0.016 inch                                                            |

Because of special interest in the superelastic behaviour of the wires, particularly the variations in Af temperature relative to the mouth temperature are an important point of investigation. Most of the wires (except for 2,6,7,11,13-15,22) showed at body temperature (36.8°C ±0.4) (Mackowiak et al., 1992) a fully austenitic phase for both batches and can be properly called superelastic (Table 4).

Problems arise with some of the wires (4-6, 10-12, 16-18, 20, 21) in determining the Af as these wires already start their R-phase transition while not yet being fully austenitic. However, if the R-phase finishing temperature is below or equal to mouth temperature, the superelastic properties are surely present, the austenitic transition being completely accomplished. Taking this in account, only the transition properties of both batches of the Heat-Activated NiTi archwire (11) at body temperature were impossible to determine.
Table 4. DSC testing: Results for each LOT at different transition phases. Wires are categorized in rectangular and round wires. For each wire and its different LOTs the transition temperatures during cooling and heating were measured. Af= austenitic finishing temperature, As=austenitic starting temperature, Mf= martensitic finishing temperature, Ms=martensitic starting temperature, Rf= R-phase finishing temperature and Rs= R-phase starting temperature. Wires 2, 14 and 15 show no transition during heating and cooling. *Some transition phases couldn’t be measured, although being present

| Wire | Material       | Heating | Cooling |
|------|----------------|---------|---------|
|      |                | Rs      | Rf      | As     | Af    | Rs    | Rf    | Ms    | Mf    |
|      |                |         |         |        |       |       |       |       |       |
| Rectangular wires: 0.016 x 0.022 inch | | | | | | | | | |
| 1    | Nitinol Heat-Activated | LOT A₁  | 5.35    | 19.71  | 30.91  | 1.95  | -42.61| -65   |
|      |                 | LOT A₂  | 5.29    | 19.9   | 33.73  | 1.67  | -42.63| -64.57|
|      |                 | LOT B₁  | 10.81   | 22.13  | 23.62  | 5.29  | -37.1 | -54.21|
|      |                 | LOT B₂  | 9.79    | 20.78  | 26.27  | 2.18  | -37.96| -54.94|
| 2    | Nitinol Classic | LOT A    | no transition |         |       |       |       |       |       |
|      |                 | LOT B    | no transition |         |       |       |       |       |       |
| 3    | Memoria        | LOT A₁  | 5.95    | 12.76  | -12.22 | 3.4   | 9.81  | 0.45  |
|      |                 | LOT A₂  | 7.38    | 16.47  | -10.11 | 7.64  | 10.55 | 1.86  |
|      |                 | LOT B₁  | 3.95    | 14.61  | -10.38 | 5.94  | 10.76 | -2.54 |
|      |                 | LOT B₂  | 3.84    | 11.5   | -11.78 | 3.48  | 8.47  | -1.97 |
| 4    | Thermal NiTi   | LOT A₁  | 20.73   | 30.46  | 7.4    | *     | 26.93 | 17.85 |
|      |                 | LOT A₂  | 22.91   | 32.12  | 5.8    | *     | 28.47 | 19.16 |
|      |                 | LOT B₁  | 15.77   | 28.94  | 10.88  | *     | 24.86 | 16.91 |
|      |                 | LOT B₂  | 14.44   | 28.38  | 11.59  | *     | 24.66 | 15.22 |
| 5    | SE Niti        | LOT A₁  | 1.5     | 22.28  | -8.88  | *     | 24.38 | -1.16 |
|      |                 | LOT A₂  | 2.84    | 19.28  | -10.61 | *     | 18.53 | -0.46 |
|      |                 | LOT B₁  | 10.92   | 19.49  | -7.96  | *     | 16.64 | 6.19  |
|      |                 | LOT B₂  | 11.98   | 20.94  | -6.54  | *     | 17.46 | 6.17  |
Table 4 continues.....

|   | Material                   | LOT A 1 | LOT A 2 | LOT B 1 | LOT B 2 | LOT A 1 | LOT A 2 | LOT B 1 | LOT B 2 |
|---|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|
| 6 | SE Triple Force NiTi       | 21.68   | 38.86   | 4.54    | *       | 36.89   | 16.01   |         |         |
|   |                            | 8.29    | 16.47   | -9.58   | *       | 13.16   | 3.05    |         |         |
|   |                            | 13.45   | 25.18   | -2.17   | *       | 29.29   | 7.33    |         |         |
|   |                            | 11.73   | 18.81   | -6.61   | *       | 18.16   | 5.09    |         |         |
| 7 | Copper NiTi 40°C           |         |         |         |         |         |         |         |         |
|   |                            | 22.49   | 36.96   | 15.66   | 2.85    |         |         |         |         |
|   |                            | 24.45   | 37.08   | 17.25   | 5.17    |         |         |         |         |
|   |                            | 15.92   | 31.69   | 11.97   | -3.46   |         |         |         |         |
|   |                            | 16.17   | 31.77   | 12.12   | -3.24   |         |         |         |         |
| 8 | Copper NiTi 35°C           |         |         |         |         |         |         |         |         |
|   |                            | 0.12    | 27.42   | 11.13   | -17.4   |         |         |         |         |
|   |                            | -0.08   | 27.64   | 10.99   | -17.98  |         |         |         |         |
|   |                            | 1.56    | 30.32   | 13.02   | -16.32  |         |         |         |         |
|   |                            | 1.52    | 29.97   | 13.44   | 15.81   |         |         |         |         |
| 9 | Copper NiTi 27°C           |         |         |         |         |         |         |         |         |
|   |                            | 9.23    | 21.23   | 1.76    | -11.84  |         |         |         |         |
|   |                            | 8.21    | 20.66   | 1.12    | -13.51  |         |         |         |         |
|   |                            | 9.28    | 21.03   | 1.61    | -11.42  |         |         |         |         |
|   |                            | 8.45    | 20.24   | 1.38    | -12.38  |         |         |         |         |
| 10| NiTi                      |         |         |         |         |         |         |         |         |
|   |                            | 6.2     | 16.33   | -12.64  | *       | 12.61   | 1.91    |         |         |
|   |                            | 6.22    | 16.79   | -12.67  | *       | 12.6    | 2.14    |         |         |
|   |                            | 7.11    | 16.53   | -12.07  | *       | 13.11   | 3.15    |         |         |
|   |                            | 7.39    | 16.92   | -11.43  | *       | 13.46   | 3.79    |         |         |
| 11| Heat-Activated NiTi arches|         |         |         |         |         |         |         |         |
|   |                            | 27.88   | 41.78   | 8.98    | *       | 38.06   | 27.32   |         |         |
|   |                            | 27.12   | 40.12   | 9.05    | *       | 36.39   | 26.64   |         |         |
| 12| Rematitan Lite            |         |         |         |         |         |         |         |         |
|   |                            | 8.01    | 30.52   | -4.95   | *       | 35.32   | *       |         |         |
|   |                            |         |         | 29.01   | 6.5     | *       | 34.47   | *       |         |
| 13| Nitanium                  |         |         |         |         |         |         |         |         |
|   |                            | 7.7     | 39.23   | 36.69   | 7.01    |         |         |         |         |
|   |                            | 5.68    | 28.26   | 35.05   | -2.62   |         |         |         |         |
### Table 4 continues.....

**Round wires: 0.016 inch**

|    | Product       | LOT A | Lot B | LOT B | LOT B |
|----|---------------|-------|-------|-------|-------|
| 14 | Nitinol Classic | no transition | no transition | no transition | no transition |
| 15 | Nitinol Superelastic | no transition | no transition | no transition | no transition |
| 16 | Memoria | 8,02 | 16,29 | -10,68 | 8,4 | 13,97 | 2,44 |
|    |                | 7,21 | 21,21 | -9,11 | * | 24,07 | 3,43 |
| 17 | Thermal NiTi | 23,34 | 30,41 | 11,83 | * | 26,79 | 19,79 | -38,47 | -66,46 |
|    |                | 19,9 | 28,62 | 11,25 | * | 25,64 | 17,79 | -36,65 | -59,95 |
| 18 | SE NiTi | 6,25 | 16,64 | -11,56 | * | 15,59 | 1,65 |
|    |                | 7,09 | 17,18 | -7,44 | * | 16,09 | 1,19 |
| 19 | Copper Ni-Ti 35°C | 5,4 | 27,37 | 11,56 | -16,21 |
|    |                | 9,49 | 29,87 | 10,37 | -10,89 |
| 20 | Ni-Ti | 9,25 | 15,12 | -11,74 | * | 11,47 | 4,38 |
|    |                | 8,78 | 13,79 | -11,85 | * | 10,47 | 4,32 |
| 21 | Rematitan Lite | 8,68 | 25,65 | -6,56 | * | 25,47 | 5,62 |
|    |                | 7,19 | 31,52 | 32,29 | 0,44 |
| 22 | Nitantium | 10,35 | 35,6 | * | 37,32 | -7,74 | * |
|    |                | * | * | * | * |
Also remarkable in Table 4, absolute batch-to-batch differences in Af (or Rf when Af was not measured) were all deviating from zero, none of them having exact the same transition temperatures, although most differences were clinically acceptable. In the testing procedure a range of deviation of 2°C has to be counted for. Therefore the authors defined significant interbatch differences as differences between two LOTs starting from 5°C. Only wire 6 (SE Triple force NiTi, 0.016 x 0.022 inch), 7 (Copper NiTi 40°C, 0.016 x 0.022 inch), 13 (Nitanium, 0.016 x 0.022 inch) and 21 (Rematian Lite, 0.016 inch) showed a remarkable interbatch difference in Af/Rf. Some measurements of one LOT couldn’t be performed as a consequence of failure in the procedure (LOT B wire 16 en wire 22), which makes it impossible to compare the batch to batch differences of these wires.

3.2 Three Point Bending Test

The results of the three point bending tests on each archwire sample are depicted as a load-deflection curve. Although the clinician has specific interest in the parameters of the unloading plateau (Fischer-Brandies et al., 2003; Pernier et al., 2005; Bartzela et al., 2007) the investigation comprised an analysis of the loading superelastic plateau using the static force (N) as a function of displacement (µm). As it is a constant, this point was only calculated for the curve with maximal deflection of 2500 µm at 22°C and 37°C and for wire 1 and 3 at 60°C and for a maximal deflection of 3000 µm for the other wires at 60°C (Fig. 2).

![Fig. 2. Superimposition of LOT A and B. This figure shows visually the interbatch difference between LOT A and LOT B of wire 7. As one can see, the two batches are quite similar and do not show a remarkable interbatch difference](image)

The obtained data from the three point bending test are summarized in Table 5. Wires 14 and 15 showed no transition for any of the fixed temperatures as seen in Table 3. Both wires didn’t show the typical deflection curve of superelastic wires (Fig. 3).
Table 5. Results from the three point bending test. Static force (N) and displacement (µm) are shown for both LOT A and LOT B of each wire. Wires 14 and 15 show no transition as seen in Table 4. These wires are linear superelastic (Fig. 5). * Wires 2, 17 and 22 (at 60ºC) were not measured due to clamping problems.

| Wire | 22ºC Static | 22ºC Displacement | 37ºC Static | 37ºC Displacement | 60ºC Static | 60ºC Displacement |
|------|-------------|------------------|-------------|------------------|-------------|------------------|
| 1    | LOT A 2,317 | 1385             | 3,18        | 1429             | 4,56        | 1941             |
|      | LOT B 2,454 | 1381             | 3,265       | 1437             | 4,517       | 1998             |
| 2    | LOT A *     | *                | *           | *                | *           | *                |
|      | LOT B *     | *                | *           | *                | *           | *                |
| 3    | LOT A 3,026 | 1752             | 3,663       | 1760             | 4,796       | 2184             |
|      | LOT B 3,02  | 1667             | 3,642       | 1756             | 4,722       | 2188             |
| 4    | LOT A 2,066 | 1876             | 2,779       | 1503             | 3,986       | 1889             |
|      | LOT B 2,054 | 1949             | 2,721       | 1490             | 3,959       | 1856             |
| 5    | LOT A 2,878 | 1726             | 3,515       | 1791             | 4,612       | 2233             |
|      | LOT B 2,684 | 1587             | 3,346       | 1669             | 4,419       | 2143             |
| 6    | LOT A 2,972 | 1603             | 3,727       | 1652             | 4,821       | 2094             |
|      | LOT B 2,992 | 1562             | 3,743       | 1653             | 4,801       | 2128             |
| 7    | LOT A 1,132 | 592              | 1,967       | 962              | 3,318       | 1559             |
|      | LOT B 1,039 | 686              | 1,968       | 893              | 3,222       | 1474             |
| 8    | LOT A 2,05  | 1084             | 2,907       | 1486             | 4,214       | 2189             |
|      | LOT B 2     | 1096             | 2,718       | 1360             | 3,928       | 1884             |
| 9    | LOT A 1,907 | 938              | 2,72        | 1291             | 3,916       | 1905             |
|      | LOT B 1,901 | 921              | 2,684       | 1267             | 3,908       | 1815             |
| 10   | LOT A 3,137 | 1665             | 3,897       | 1803             | 4,856       | 2254             |
|      | LOT B 2,85  | 1896             | 3,617       | 1746             | 4,655       | 2221             |
| 11   | LOT A 2,374 | 2319             | 2,893       | 2022             | 4,133       | 1896             |
|      | LOT B 2,281 | 2236             | 2,834       | 2934             | 4,128       | 4128             |
| 12   | LOT A 3,033 | 2169             | 3,665       | 1945             | 4,746       | 2148             |
|      | LOT B 3,67  | 2197             | 4,301       | 2051             | 5,292       | 2323             |
| 13   | LOT A 2,954 | 2239             | 3,646       | 1995             | 4,809       | 2081             |
|      | LOT B 3,361 | 2441             | 4,105       | 2526             | 5,194       | 2502             |
| 14   | LOT A /     | /                | /           | /                | /           | /                |
|      | LOT B /     | /                | /           | /                | /           | /                |
| 15   | LOT A /     | /                | /           | /                | /           | /                |
|      | LOT B /     | /                | /           | /                | /           | /                |
| 16   | LOT A 1,164 | 2530             | 1,433       | 2644             | 1,636       | 2847             |
|      | LOT B 1,045 | 2388             | 1,296       | 2465             | 1,66        | 2916             |
| 17   | LOT A *     | *                | *           | *                | *           | *                |
|      | LOT B *     | *                | *           | *                | *           | *                |
| 18   | LOT A 0,897 | 2676             | 1,216       | 2428             | 1,59        | 2977             |
|      | LOT B 0,827 | 2311             | 1,107       | 2347             | 1,482       | 2859             |
| 19   | LOT A 0,604 | 1340             | 0,9748      | 1868             | 1,523       | 2778             |
|      | LOT B 0,405 | 759              | 0,769       | 1336             | 1,378       | 2209             |
| 20   | LOT A 1,002 | 2461             | 1,361       | 2473             | 1,806       | 2899             |
|      | LOT B 1,023 | 2587             | 1,375       | 2534             | 1,769       | 2891             |
| 21   | LOT A 0,955 | 2724             | 1,312       | 2503             | 1,7         | 2781             |
|      | LOT B 1,029 | 2766             | 1,346       | 2665             | 1,723       | 3010             |
| 22   | LOT A 1,179 | 3172             | 1,537       | 3179             | *           | *                |
|      | LOT B 1,011 | 3187             | 1,333       | 3183             | *           | *                |
These wires can be defined as linear (super) elastic wires (Zadno and Duerig, 1990). They typically exhibit a nearly hysteresis-free linear pseudoelasticity with elastically recoverable strains as high as 4%. The deformation of these cold-worked NiTi wires is not controlled by a stress-induced martensitic transformation, as observed in a conventional superelastic material. This difference can be of significant potential advantage because of the temperature and compositional independence of its mechanical properties (Zadno and Duerig, 1990). Due to mechanical cycling in the end, two parameters (unresolved strain and hysteresis) of the cold-worked NiTi alloy decrease, which is obviously depicted in Fig. 3.

![Wire 15](image)

**Fig. 3.** Load-deflection curve of wire 15, showing no superelastic plateau at none of the fixed temperatures. A typical load-deflection curve of a linear superelastic NiTi alloy shows no flat area and the load is nearly proportional to the increasing and decreasing deflection, which can be defined as a small hysteresis. Due to mechanical cycling in the end the unresolved strain and hysteresis decrease.

Also for the three point bending measurements the mechanical properties at 37ºC are of special interest from a clinical point of view. As derived from Table 4 none of the compared batches for each wire showed superelasticity at exactly the same point.

For a better overview the interbatch differences for both parameters displacement and static force representing the superelastic point are visualized in Fig. 4. For every LOT the absolute difference in superelasticity between the batches was divided by the mean of the two batches and expressed in percentages deviating from the mean.

Remarkable interbatch differences (>10% deviation to the mean for at least one of the parameters) were seen in 5 wires (11, 12, 13, 19, 22) of all 22 tested samples. Wires 19 and 13 obviously deviate for displacement as well as static force variations. Wires 12 and 22...
deviated more than 10% only for static force differences, whether wire 11 has a deviation of more as 35 % what’s concerning displacement difference.

The aim of this study consisted also of a survey of the superelastic properties of each archwire. Figs. 5 and 6 show the percentage of superelasticity as a function of deflection up to 3500 µm at a temperature of 37°C. For almost all tested 0.016 x 0.022 NiTi archwires a non-superelastic curve was seen below a deflection of 1500 µm, except for wire 7 en wire 9 for both batches having the superelastic properties at smaller displacements (Fig. 5).

For the round 0.016 NiTi archwires superelasticity was reflected only at a minimum deflection of 2500 micrometer, apart from wires 18 and 19 (Fig. 6).

Thus in most situations the elastic properties of superelastic NiTi wires might be based on linear elastic models, which bring us to the question that was stated earlier. Small deformations do not activate this superelastic plateau and only a linear elasticity is performing the unloading force on the teeth, not being constant (Brantley et al., 2001; Guénin, 1986).
3.3 Scanning Electron Microscopy

The results are summarized in Table 6. Most of the round wires have a smooth surface and only one wire has longitudinal grooves. Wires 6, 8 and 9 show a remarkable interbatch difference. Lot B of wire 6 and LOT B of wire 8 display a smooth surface, however LOT A
shows a rough surface (Fig. 9). For wire 9, the opposite is seen. Other wires show no remarkable difference between their batches (Figs. 7 and 8 and 12).

Table 6. Scanning electron microscopy results. If there is an interbatch difference of the surface topography each LOT was categorized separately in the table. The absence of interbatch differences are marked with an x

| Wire | Smooth surface | Longitudinal grooves | Rough surface | Other |
|------|----------------|----------------------|--------------|-------|
| Rectangular wires: 0.016 x 0.022 inch | | | | |
| 1    | X              |                      |              |       |
| 2    | X              |                      |              |       |
| 3    | X              |                      |              |       |
| 4    | X              |                      |              |       |
| 5    | X              |                      |              |       |
| 6    | LOT B          |                      | LOT A        |       |
| 7    | X              |                      |              |       |
| 8    | LOT B          |                      | LOT A        |       |
| 9    | LOT A          |                      | LOT B        |       |
| 10   | x              | (defects)            |              |       |
| 11   | x              | (crisscross grooves) |              |       |
| 12   | x              | (defects)            |              |       |
| 13   | x              | (defects)            |              |       |
| Round wires: 0.016 inch | | | | |
| 14   | X              |                      |              |       |
| 15   | X              |                      |              |       |
| 16   | x              |                      |              |       |
| 17   | x              |                      |              |       |
| 18   | LOT B          |                      | LOT A        |       |
| 19   | x              | (defects)            |              |       |
| 20   | x              |                      |              |       |
| 21   | x              |                      |              |       |
| 22   | x              |                      |              |       |

Fig. 7. Wire 3: No interbatch difference: smooth surface. A) left = LOT B, right = LOT A and b) detail LOT A
Most of the rectangular wires also have a smooth surface. One wire displays a rough surface in both batches (Fig. 8) and two wires have longitudinal grooves (Fig. 11). The interbatch differences were seen in only one wire (G&H wire company). LOT A shows defects in the wire while LOT B has a smooth surface (Fig. 10).

Fig. 8. Wire 19: No interbatch difference: rough surface. a) left = LOT B, right = LOT A and b) detail of LOT A

Fig. 9. Wire 8: Remarkable interbatch difference: a) LOT A shows a rough surface in comparison with LOT B (left = LOT B, right = LOT A), b) detail of LOT B and c) detail of LOT A

Fig. 10. Wire 18: Remarkable interbatch difference: LOT A shows surface defects. a) left = LOT B, right = LOT A and b) detail of LOT A
3. DISCUSSION

Superelastic wires have been widely used in clinical orthodontics due to their high springback and low stiffness. Selection of an appropriate wire for a specific clinical condition becomes very important. It is known that wires have to produce constant forces over different deflection rates and that the clinician must be fully aware of the exact force delivery of the chosen wire.

It is also clear that one cannot easily transfer laboratory findings of the previous testing procedures to a clinical orthodontic setting. All testing results therefore have to be carefully interpreted. The laboratory findings should be compared to clinical trials to reveal important characteristics of the archwires (Bartzela et al., 2007).

The transformation temperatures of nickel-titanium wires are critical factors in their transformation behaviour. In the current investigation especially Af is of great importance.

Several experiments have been performed in the past to verify whether the TTR of the archwires correspond to the values provided by the manufacturers. It is clear that the TTR of
the alloy should be close to or slightly below the mouth temperature to allow the transformation. The alloy phase structure is a function of mechanical stress and environmental temperature (Fischer-Brandies et al., 2003).

Different studies indicated that alterations in mouth temperature due to intake of cold or hot liquids or food could cause a stress fluctuation in NiTi wires during orthodontic treatment (Airoldi et al., 1997; Meling and Ødegaard, 2001). Previously this was believed to exert only transient effects. But a recent study of Meling and Ødegaard, 2001 indicated that some effects could be prolonged. They tested three NiTi wires to study the effect of short-term temperature changes on bending stiffness. The samples were tested in both activation and de-activation phase. They concluded that the bending stiffness of some superelastic NiTi wires can be markedly affected for a prolonged period of time by short-term temperature changes.

As mentioned earlier light continuous forces are seen as ideal in an orthodontic treatment (Daskalogianakis and McLachlan, 1996; Darendeliler et al., 1997; Owman-Mol et al., 1995). An experimental study, however, showed that application of discontinuous forces promotes tissue repair during unloading and results in less root resorption than application of continuous forces (Acar et al., 1999).

The batch-to-batch differences found in this study do confirm previous findings. Langeron-Gardrinier (1999) subjected two batches of two NiTi coils to DSC, the measurements being different for the ORMCO as well as for the GAC supplied coils.

Interbatch variations especially become critical if their transition temperatures differ in a way that two LOTs have a different phase structure (wires 7,13,21) at mouth temperature (37°C), exercising clinically various mechanical properties.

The results of the 0.016 inch NiTi Ormco wire (10) in our study differs from the research by Bradley et al. (1996) who found in his experiments that the given wire was almost entirely austenite at body temperature, in contrast to its already full austenitic structure at room temperature in our findings. The clinical results of Rematitan Lite (12,21) and Copper NiTi 35°C (8,19) are in accordance with the study of Fischer-Brandies et al. (2003).

Interbatch differences of the mechanical properties do confirm other findings with NiTi coils, being comparable to NiTi wires, where the force systems of the springs were strongly influenced by the alloy and the batch under investigation (Bourauel et al., 1997).

An important source of variation in NiTi experiments is the complexity of the manufacturing process. Variations in the mechanical and thermal properties of NiTi alloys are shown to be affected by their chemical composition and by the process of fabrication.

The production process consists of high vacuum melting, press forging, rotary swagging and rod/wire rolling (Degarmo et al., 2003; Smith and Hashemi, 2003). The double vacuum melting manufacturing process ensures purity and quality and maintains the mechanical properties of the alloy. The raw materials are carefully formulated before the alloy is melted by vacuum induction. After this process, vacuum arc remelting takes place to improve the alloy chemistry, homogeneity and structure (Thompson, 2000).

Once the alloy has been manufactured, it undergoes various processes before being a finished arch wire. Essentially, the casting is forged in a press into a cylindrical shape prior to
rotary swaging under pressure, to create a drawn wire. The wire is then rolled to form a tapered shape with even pressure from a series of rollers applied to the wire.

During this processes, the alloy is subjected to heat-treatments. Heat-treatment of NiTi alloys is delicate and critical. It involves recovery annealing after cold working. It is the essential tool in fine-tuning the transformation temperatures. Aging time and temperature control the precipitation of various Ni-rich phases and the dislocation arrangements. Precipitation thus controls how much nickel residues on the NiTi lattice; by depleting the matrix of nickel, aging increases the transformation temperature. Therefore, the combination of cold working followed by appropriate heat-treatment is essential in controlling the properties of Nitinol (Fischer-Brandies et al., 2003; Degarmo et al., 2003; Pelton et al., 2003; Saburi et al., 1982; Mercier and Torok, 1982). For example, the position of NiTi wires in the furnace whilst heat-treatment could determine the thermal and mechanical properties. Indeed, the temperature of the oven can slightly differ at various places from the beginning till the end of heat-treatment (Mariot, 2001). These kinds of manufacturing inaccuracies could explain the batch-to-batch differences found in this study. Wires of similar composition can differ strongly especially when they are manufactured by different suppliers. As a result, the findings of experimental studies are hardly comparable. This could explain the quantitative differences between LOTs of the same wire and manufacturer found in this study.

Moreover, some manufacturers provide accurate information about the properties of their NiTi wires, whereas others do not (Santoro et al. 2001). Deriving from non-martensitic transformation DSC curve was expected for the Nitinol Classic archwires 2 and 14 as those archwires have a martensite stabilized crystal structure. However, for the Nitinol SE (15), more appropriate consumer information should be given as the term superelastic in this case is not inappropriate.

Apart from some manufacturing inaccuracies, measurement errors/bias should be taken in account too.

Moreover, as it comes to batch-to-batch differences, caution should be taken in drawing conclusions. First, only one sample from each batch was compared to another, so no relevant statistics could be done and second, for those wires where two measurements for every LOT were obtained (1 and 3-10) with the DSC procedure, the absolute differences (Table 7) are comparable to the mean differences between the different LOTs (data on request). The temperature measurement as performed with the DSC reveals a measurement error in the order of 1 to 2ºC. Comparison with other studies could be directionally to this measurement error.

| N  | mean | Std  | Max  | Q3  | Median | Q1  | Min   |
|----|------|------|------|-----|--------|-----|-------|
| 88 | 2.620| 5.280| 32.130| 2.150| 0.990  | 0.320| 0.010 |

Although some of the wires deviate more in absolute difference than others concerning the mechanical properties, clinical significant differences could hardly be given. After all, one could ask himself if such minimal differences in applied forces to the teeth would influence tooth movement, especially in the starting phase of leveling and alignment. In a recent study
of van Leeuwen et al. (2010). The rate of orthodontic tooth movement was evaluated by altering forces during bodily tooth movement in eight beagle dogs. Forces of 10 or 300 cN were randomly assigned to each side of the dogs. They stated that a positive dose-response relation only exists in a very low force range. In higher force ranges, they could not establish such relation.

Ren et al. (2003) tried to evaluate the relation between force magnitude and rate of tooth movement. They carried out a review of the optimal force magnitude in orthodontic tooth movement. He concluded that it was not possible to perform a meta-analysis from the current literature, so no evidence-based force level could be recommended for optimal efficiency. One year later Ren et al. (2004) tried to develop a mathematical model to describe the relationship between magnitude of applied force and rate of orthodontic tooth movement. They could not determine a threshold value that would switch on tooth movement. The model showed a wide range of forces all leading to a maximum rate of tooth movement.

However, a clinically more important finding is the next: for most of the round wires only full expression of the superelastic property occurred in cases with severe crowding (2500 micrometer displacement).

Most of the rectangular wires had a better score: already light continuous forces were exercised beginning at a crowding of 1.5 mm contact point displacements. This is a clinical important finding as we use rectangular NiTi wires when little crowding remains. However, at small deformations, most of the alloys show a linear elasticity, and only at larger deformations above a given value, the martensitic transformation occurs.

The SEM images of each sample revealed a significant difference in finishing quality of the surface of different orthodontic wires. This difference was the larger when different suppliers were compared, although there was also a difference between batches.

Surface differences are relevant since the quality of the surface is very important in friction between the arch and the bracket but also in contamination and corrosion resistance (Fischer-Brandies et al., 2003).

4. CONCLUSION

This study clearly shows the presence of batch-to-batch differences, which is why the selection of archwires constitutes an important aspect of orthodontic treatment.

CONSENT

Obtaining informed consent from patients was not applicable since the present study consisted of pure in vitro material research independent from any clinical patient activity.

ETHICAL APPROVAL

Obtaining ethical approval was not applicable in view of the fact that the present study concentrated on comparing mechanical and thermal properties of different batches of a great number of orthodontic wires in vitro.
COMPETING INTERESTS

Authors have declared that no competing interests exist.

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