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Torsional properties of Ni–Ti and Copper Ni–Ti wires: the effect of temperature on physical properties

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SUMMARY The stress–strain behaviour of two Ni–Ti and two Copper Ni–Ti orthodontic wires was examined in induced torsion under controlled conditions of moment and temperature. The tests clearly demonstrate the diversity of behaviour of these wires. The loading and unloading curves and plateau regions were found to be closely related to temperature with stiffness varying dramatically over mouth temperature range under identical stress. Diversity of reaction to stress is linked to the crystalline structure of the alloys.

Introduction

Nickel–titanium (Ni–Ti) wires were first introduced for orthodontic use in 1972 under the brand name of Nitinol (acronym of Nickel Titanium Naval Ordnance Laboratory). In 1985, a new Ni–Ti alloy was developed in China by the Non Ferrous General Institute of Beijing (Burstone *et al.*, 1985), in 1986 a Ni–Ti was commercialized in Japan (Miura *et al.*, 1986) and more recently a Copper Ni–Ti has been launched in the USA.

When an ordinary alloy is stressed beyond its elastic limit, the resulting deformation remains even after the mechanical stress has been removed. Some alloys, on the other hand, particularly the Ni–Ti alloys, can return to their former shape when the initial conditions are reapplied. This phenomenon is known as superelasticity (Miura *et al.*, 1986) and is used in orthodontics to initiate tooth movement.

Superelasticity is linked to structural changes within the material and takes place at certain temperatures. The structural state at high temperature is called 'austenite', and that at low temperature 'martensite'. Such a transformation is called 'martensitic'. It is reversible when temperatures vary and occurs without diffusion of the nickel and titanium. However, another transformation can also occur between the austenitic and martensitic phases known as the 'R-phase' (Miyazaki and Otsuka, 1986). The R-phase has a rhombohedral symmetry and a simple hexagonal lattice. The space group of this structure is $P\bar{3}1m$. The A → R transformation is first-order with an enthalpy of transformation between 3 and 4 J/g (Goo *et al.*, 1985). A shape memory effect and pseudoelasticity associated with the austenite → R-phase transition can be observed. Structural changes with cooling and heating of Ni–Ti alloys can involve three phases: austenite, R-phase and martensite. On cooling, the sequence is:

- either AUSTENITE → MARTENSITE and the sequence on heating MARTENSITE → AUSTENITE
- or AUSTENITE → R-PHASE → MARTENSITE and the reverse transformation from martensite, on heating, can be associated with one or two transformations MARTENSITE → AUSTENITE or MARTENSITE → R-PHASE → AUSTENITE (Jordan *et al.*, 1995).

The corresponding transformation sequences can be usefully studied by differential scanning calorimetry (DSC). DSC is a general technique which measures the enthalpy of a transition, which in this particular experiment is the martensitic transition. Structural transformation is identified by a peak in the studied property versus temperature plot. DSC provides rapid

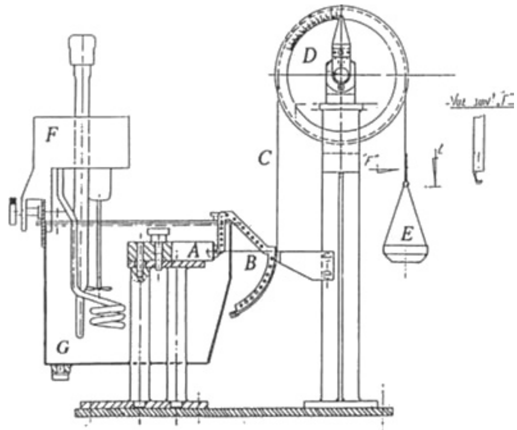

Figure 1 Diagrammatic representation of testing bench. A, dental simulator; B, lever; C, strip of iron; D, graduated pulley; E, weight; F, thermostatic bobbing heater; G, container (Filleul, 1989).

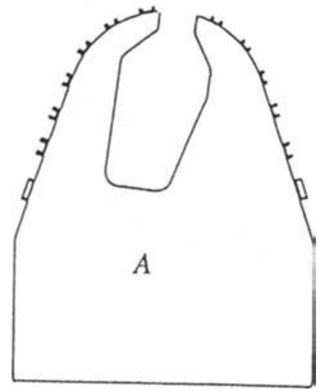

Figure 2 Dental arch simulator (Filleul, 1989).

information on transformation temperatures (Jordan *et al.*, 1994). The R transformation can be improved by the addition of a third element (such as Co, Fe) or by thermomechanical treatments.

Such transformation temperatures are highly sensitive to the chemical composition and to the thermomechanical history of the studied alloy (Guenin, 1986). As far as ordinary alloys are concerned, the strain is proportional to the applied stress (below the elastic limit) and the elastic modulus is constant. Stress-strain curves are approximately linear.

On the other hand, the elastic modulus of Ni–Ti alloys is not constant and the stress-strain curves exhibit typical plateaux linked to structural changes within the material.

It is, therefore, necessary to test such Ni–Ti alloys under experimental conditions reproducing as closely as possible the true intra-oral clinical environment (Burstone *et al.*, 1985).

Few authors have studied orthodontic wires made from these alloys in bending (Tonner and Waters, 1994) and no torsion experiments have been reported, despite the fact that the behaviour of wires under stress induced by torsion is considerably different from their behaviour under stress induced by flexion. This is due to the fact that the distance between orthodontic brackets influences the stiffness of the archwire differently when flexion or torsion is applied: indeed, the inter-bracket distance follows a third power law in flexion, whereas in torsion the effect on the distance is linear.

The purpose of this study was to test Ni–Ti and copper Ni–Ti orthodontic wires, in torsion at temperatures of 22, 39 and 44°C, in order to approach normal clinical conditions. DSC was used to identify the crystallographic phases of alloys at various temperatures (Jordan *et al.*, 1994).

Materials and methods

An original testing bench was designed and set up to test orthodontic wires in induced torsion under controlled conditions of moment and temperatures reproducing as closely as possible the intra-oral environment (Filleul, 1989).

The test apparatus is shown in Figure 1. Torsion is applied to the system by adding loads in increments of 1 g to the scale pan (E) which is connected via a graduated pulley (D) to a lever (B). The design is such that the centre of resistance of the lever, defined by the position of the archwire, remains constant during the test. The radius of curvature of the pulley and lever are identical, enabling the archwire torsion, in degrees, to be read directly on the pulley. The moment (M) applied to the archwire is given by the product of the distance from the axis of the wire to the plane of applied force and the force itself.

$$M = F \times d$$

Table 1 Details of the various archwires used.

Material	Nominal section (inches)	Manufacturer/ supplier
US Ni–Ti® Broad arch. Upper/Small	0.017 × 0.025	Ormco Corp., Glendora, CA, USA
Neo Sentalloy F100® Accu Form	0.017 × 0.025	GAC, International, made in Japan
Copper Ni–Ti 35® Broad arch. Upper/Small	0.017 × 0.025	Ormco Corp., Glendora, CA, USA
Copper Ni–Ti 40® Broad arch. Upper/Small	0.017 × 0.025	Ormco Corp., Glendora, CA, USA

The test wire is attached to the simulator (A) (Figure 2) which has the form of a dental arch with the upper right central incisor missing.

The wire is tied into 0.018 × 0.025 inch brackets neither tipped nor angulated (central: GAC W105-18, lateral: GAC W104-18, canine: GAC W108-18, premolar: GAC W108-18, first molar: GAC W106-18, left second molar: GAC W8100-L, right second molar: GAC W8100-R) with elastomeric ligatures (GAC 110 clear) (GAC International Inc., NY, USA). At the beginning of the experiment, there is a certain amount of 'slack' in the apparatus which must be taken up before torsion is applied, and following the work of Creekmore (1979) the first 4.5 degrees of torsion were ignored.

The system is immersed in a thermostatically controlled waterbath, and measurements were made at 22, 39 and 44°C. These temperatures were selected to encompass the range encountered intra-orally.

Four different brands of Ni–Ti alloy 0.017 × 0.025 inch orthodontic archwire, for which superelastic capabilities were claimed, were selected. Five archwires drawn from the same commercial batch for each brand were tested in torsion to evaluate the true amount of restored torque (Table 1).

Each wire was loaded with moments varying from 0 to 1400 g.mm and unloaded with moments varying from 1400 to 0 g.mm, successively at 22, 39 and 44°C, and the mean values of the five measurements made on the archwires for each brand were plotted on a graph.

All wires were commercially available as preformed archwires. Their specifications are given in Table 1. The exact composition and thermomechanical treatment of these wires is unknown.

DSC was used to characterize the Ni–Ti wires. A Mettler 30/TA 4000 apparatus (Mettler-Toledo AG, Im Langacher, CH-8606 Greinfensee, Switzerland) was used at a cooling/heating rate of 5°C/min. The spectra obtained on heating or cooling show peaks related to the transformation phenomena. These were exothermic on cooling, and endothermic on heating.

Results

The measured deformation is shown in Figures 3–9. This allows the following propositions to be made.

The US Ni–Ti® (Figure 3) showed small differences between the moments registered at 39 and 44°C. On the other hand, this wire was less stiff at the lower temperature of 22°C. Between 70 and 1400 g.mm, the loading and unloading curves were linear. The shapes of the curves at all temperatures were identical. There was no sign of the plateau typical of the superelastic effect and thus a true elastic property seems to exist.

Neo Sentalloy F100® (Figure 4) showed few observed differences between restoring torques at 39 and at 44°C. However, the stiffness of Neo Sentalloy F100® decreased at 22°C. At that temperature, the plateau typical of the superelastic effect was apparent up to a threshold of stress of 910 g.mm. In the plateau region, the force applied to a tooth would therefore be almost constant.

Copper Ni–Ti 35® (Figure 5) showed different loading and unloading curves. The restoring torques delivered at 22°C were lower (as was the case with the US Ni–Ti® and Neo Sentalloy F100®) than at 39 and at 44°C. At 22 and at 39°C, the observed curves were typical of superelasticity, but not identical. At 22°C, the Copper Ni–Ti 35® maintained a certain deformation (27.8 degrees torsion). On the other

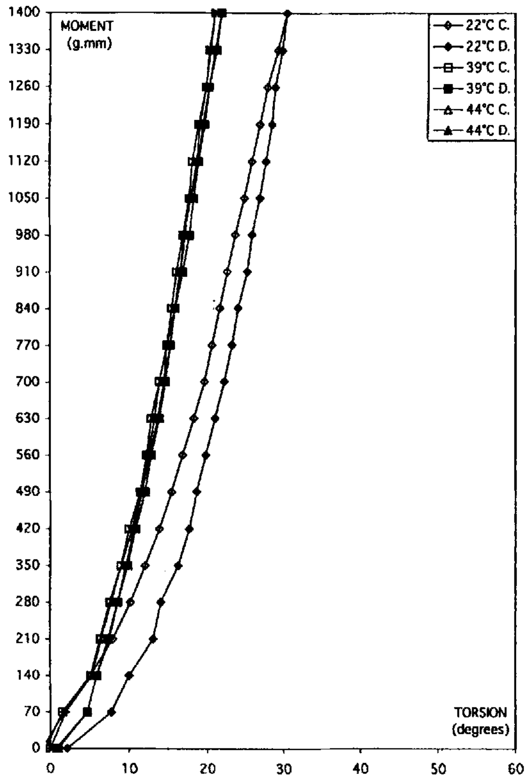

Figure 3 Curves of US Ni–Ti®, C = loading, D = unloading.

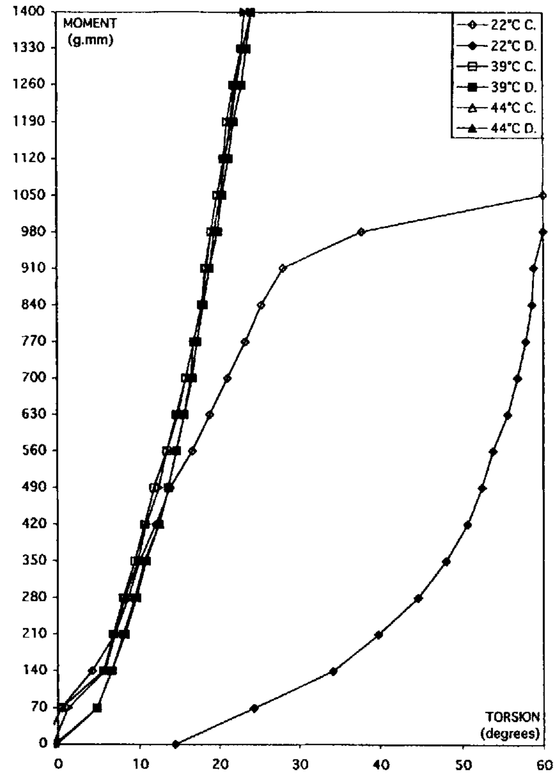

Figure 4 Curves of Neo Sentalloy F100®, C = loading, D = unloading.

hand, the superelastic effect was complete at 39°C. At the end of unloading, the Copper Ni–Ti 35® returned to its initial shape (0 degrees torsion). The plateau typical of the superelastic effect seems to appear for a moment of 560 g.mm at 22°C, and for a moment of 1190 g.mm at 39°C. At 44°C, the loading–unloading curve was linear with a slope characteristic of an austenite elastic modulus. At such a higher temperature, there was no transformation within the alloy. True elasticity was thus responsible for the return of the deformed archwire to its original shape, with the induced tooth movement required by orthodontists.

Copper Ni–Ti 40® curves (Figure 6) were similar to Copper Ni–Ti 35®. At 44°C, loading and unloading curves were linear, as observed with Copper Ni–Ti 35®. Here again, it is the elasticity of the wire which is at work. At 22 and at 39°C, the observed curves were typical of superelasticity, as for Copper Ni–Ti 35®. Differential behaviours nevertheless appeared between the two alloys, during the unloading phase. At 39°C, the plateau of the curve typical of superelasticity appeared with a delivered moment of 560 g.mm with Copper Ni–Ti 40®, considerably less than with Copper Ni–Ti 35® (700 g.mm).

Superimposition of the four curves, each corresponding to one of the different wires under test, clearly show the variety of their behaviour at 22, 39 and 44°C. Figures 7–9 demonstrate the load/deflection behaviour of the wire when exposed to temperatures very close to the conditions in clinical use.

Testing the wires at 22°C (Figure 7)

US Ni–Ti® alone showed no superelastic effect.

Testing the wires at 39°C (Figure 8)

Both US Ni–Ti® and Neo Sentalloy F100®

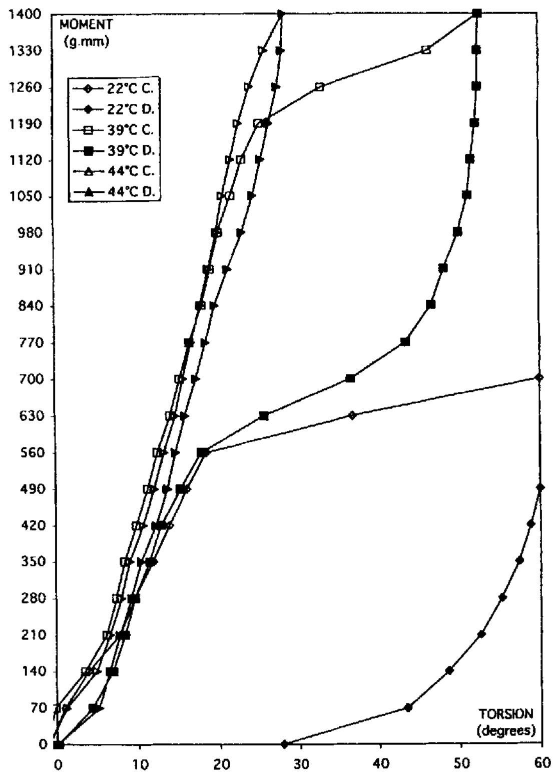

Figure 5 Curves of Copper Ni–Ti 35®, C = loading, D = unloading.

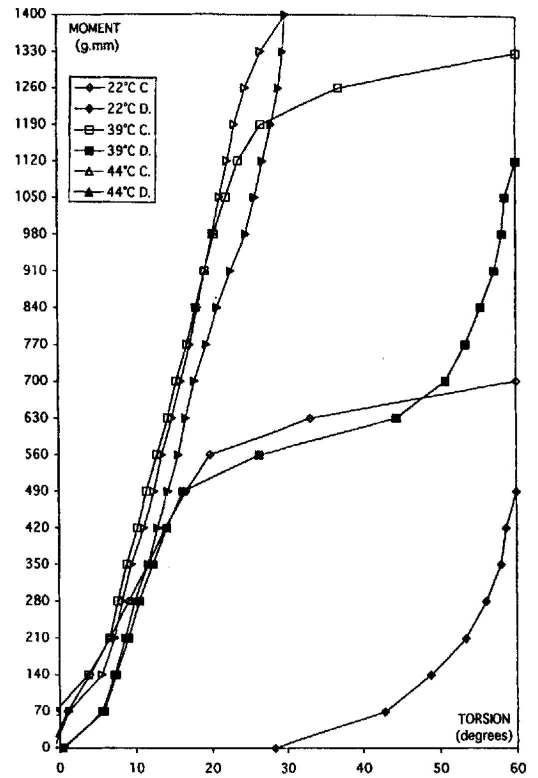

Figure 6 Curves of Copper Ni–Ti 40®, C = loading, D = unloading.

showed no superelastic effect, whilst the two other brands showed superelastic effects.

Testing the wires at 44°C (Figure 9)

None of the brands of wire tested showed any superelastic effects. It is essentially the elasticity of the wire which is used.

This first series of tests clearly demonstrated the diversity of behaviour of wires furnished by the different makers under identical amounts of stress, as environmental temperatures increase.

Differential Scanning Calorimetry

Diversity of reaction to stress might be linked to the crystalline structure of the alloys, and this was investigated using DSC. DSC measures four temperature thresholds which explain the diversity of behaviours of Ni–Ti and Copper Ni–Ti orthodontic wires (Table 2).

During the cooling phase, Rs is the temperature threshold under which the R-phase always appears, even when no mechanical stress is applied, and Ms is the temperature threshold below which the martensitic phase always appears, even when no mechanical stress is applied.

During the heating phase, Rf′ is the temperature beyond which the R-phase always reverts to austenitic phase, when no mechanical stress is applied (Table 2), and Af the temperature beyond which the martensitic phase always reverts to austenitic phase, when no mechanical stress is applied.

US Ni–Ti®

DSC shows that during cooling US Ni–Ti® undergoes austenitic transformation into R-phase (Rs) at 31.7°C (Table 2), and explains why, at 39 and at 44°C, the behaviour of this alloy at these two temperatures remains similar. At 22°C, the R-phase was achieved in the US Ni–Ti® orthodontic wires. The slope of the curve at 22°C was lower than the slope of the curves at 39 and at 44°C (Figure 3). This is consistent with

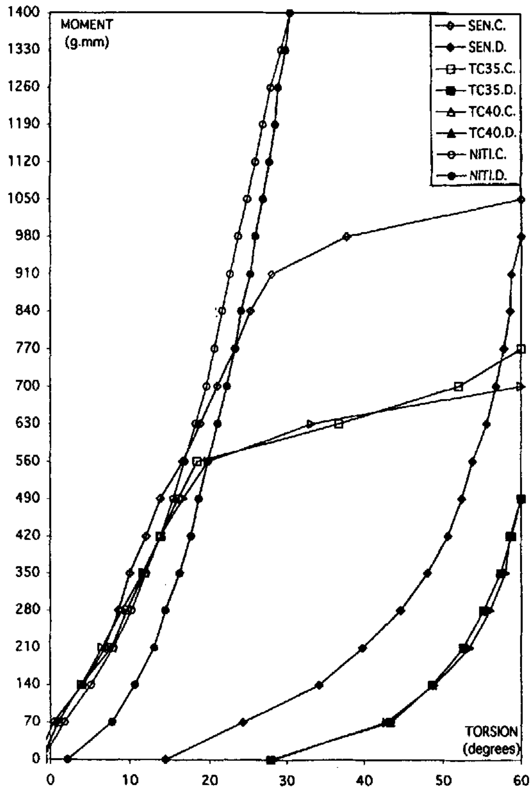

Figure 7 Superimposition at 22°C, C = loading, D = unloading.

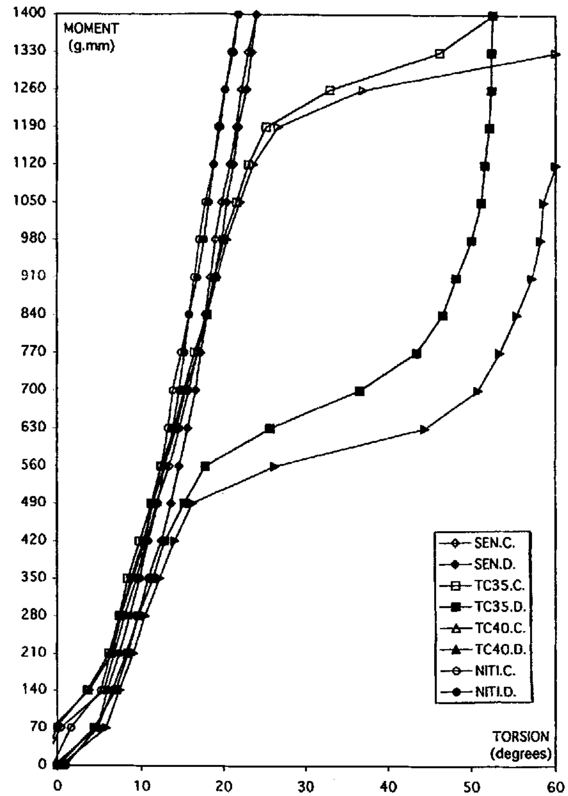

Figure 8 Superimposition at 39°C, C = loading, D = unloading.

the known fact that the R-phase has an elastic modulus very much lower than the austenitic phase (Tonner *et al.*, 1994). For the same amount of torsion of an edgewise wire, the developed moments at 22°C will be weaker than those at 39 or at 44°C.

At the levels of temperatures used in clinical orthodontics (22–44°C), and with the amounts of stress sufficient to induce tooth movement (0–60 degrees of edgewise torsion), no super-elastic effect was shown by the US Ni–Ti® (Figure 3).

Neo Sentalloy F100®

DSC shows that Neo Sentalloy F100® during cooling undergoes austenitic transformation into R-phase (Rs) at 21.7°C (Table 2) and remains completely austenitic at 22, 39 and at 44°C. When the Neo Sentalloy F100® undergoes stress at 22°C, a linear elastic strain is first observed (Figure 4). As the strain increases, a plateau appears at 910 g.mm. When the stress stops, it reverts almost to its initial shape, and simple heating will completely restore its former shape.

The really important point is that the transformation temperature from the austenitic phase to the R-phase is 21.7°C. At 39 and 44°C, the alloy remains austenitic and the amounts of stress applied experimentally remain insufficient to induce an R-transformation. The registered results under stress by torsion applied to orthodontic wires express the elastic modulus of the austenite (this is consistent with the observation that the curve remains linear). On the other hand at 22°C (temperature very close to Rs = 21.7°C), the mechanical energy gradually applied during the loading phase was sufficient to allow the R-phase to appear. The orthodontist can thus use the superelasticity of the trans-

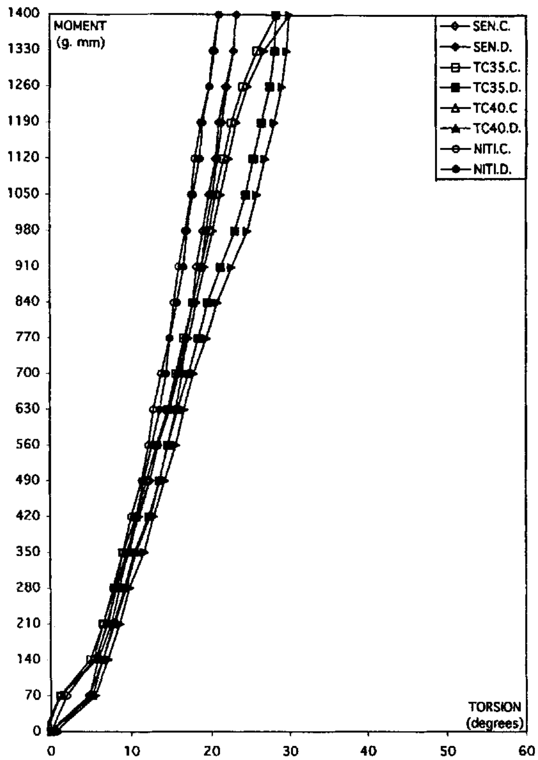

Figure 9 Superimposition at 44°C, C = loading, D = unloading.

formation (austenite to R-phase) to engage the wire in the slot of the bracket safely without the risk of bond failure.

During unloading, the R-phase present creates a stress/strain ratio smaller than when the wire is used with austenite. The moments developed on the teeth are of smaller magnitude. Moreover, at the end of the treatment the wire resumes its initial shape (0 degrees torsion) because the transformation temperature of R-phase into austenite (Rf′) = 27°C is smaller than buccal temperature (Table 2).

The DSC of Copper Ni–Ti 35® while cooling shows that the austenitic transformation into martensite (Ms) occurs at 17.5°C (Table 2). Copper Ni–Ti 35® is characterized by a direct transformation from austenite to martensite. There is no R-phase. At 22, 39 and 44°C, the alloy is austenitic.

At 22°C (Figure 5), a load of 560 g.mm is sufficient to allow the martensitic phase to

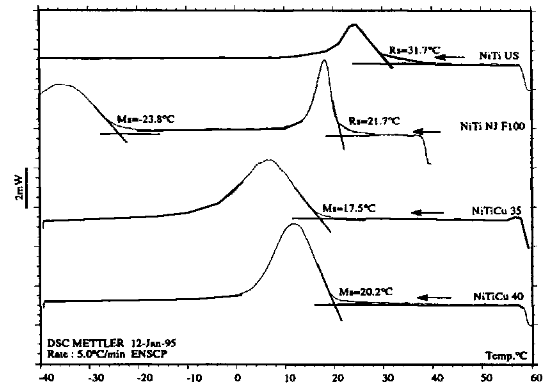

Figure 10 Superimposition of the DSC curves on cooling.

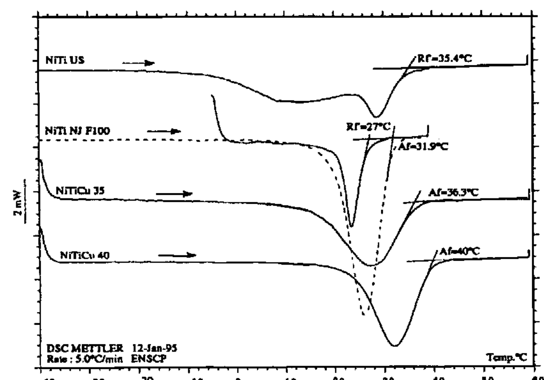

Figure 11 Superimposition of the DSC curves on heating.

Table 2 Transformation temperatures determined by DSC.

Material	Cooling		Heating	
	Rs (°C)	Ms (°C)	Rf′ (°C)	Af (°C)
US Ni–Ti®	31.7	–36.4	35.4	
Neo Sentalloy F100®	21.7	–23.8	27	
Copper Ni–Ti 35®		17.5		36.3
Copper Ni–Ti 40®		20.2		40

appear and therefore for the wire to develop superelastic behaviour since 22°C is a temperature very close to Ms = 17.5°C.

Under experimental conditions at a constant temperature of 22°C, after unloading, a degree of residual deformation remains: 27.8 degrees in torsion. This would not be the case intra-orally, with the wire returning to 0 degrees torsion, since its transformation from martensite into austenite temperature (Af) is 36.3°C (Table 2). The Copper Ni–Ti 35® can be placed very easily in the mouth without the risk of bond failure. At buccal temperature, unloading is of a smaller magnitude, and constant down to 0 degrees torsion. At 39°C (Figure 5), a load of 1190 g.mm is the smallest mechanical energy which will activate the superelasticity.

A temperature of 44°C is too far from austenitic transformation into martensite (Ms) = 17.5°C to allow supplied mechanical energy (1400 g.mm) to activate the superelasticity. It is the elasticity of austenite which is used. A higher amount of stress may possibly allow the martensitic phase to appear.

Copper Ni–Ti 40®

DSC shows that Copper Ni–Ti 40® while cooling undergoes austenitic transformation into martensite (Ms) at 20.2°C (Table 2). The alloy therefore remains austenitic (at 22, 39 and 44°C). R-phase never occurs: Copper Ni–Ti 40® being characterized by direct transformation from its austenitic to its martensitic phases.

At 22°C (Figure 6), a load of 560 g.mm (identical to the load used in the former experiment with Copper Ni–Ti 35®) also shows superelastic behaviour since 22°C is a temperature very close to Ms temperature (20.2°C). This means that Copper Ni–Ti 40® orthodontic wires can be engaged in the brackets very easily at mouth temperature without the risk of bond failure, as was observed with Copper Ni–Ti 35® wires.

At 22°C, after unloading, 28.2 degrees in torsion remain as a permanent deformation. Copper Ni–Ti 40® would only revert to 0 degrees torsion when and if the patient has a hot drink, at more than 40°C, which is the transformation temperature Af of this alloy from martensite into austenite (Table 2).

At 39°C (Figure 8), a load of 1190 g.mm will be required for superelasticity to appear, as in the case of Copper Ni–Ti 35®; the restoring torques delivered by Copper Ni–Ti 40® being nevertheless weaker than those observed with the latter.

At 44°C, as was previously observed with Copper Ni–Ti 35®, the temperature is too distant from Ms (20.2°C) to allow the maximal supplied mechanical energy tested in this experiment (1400 g.mm) to induce the appearance of superelasticity, linked to the appearance of martensite. The elasticity due to the austenitic phase of the alloy is at work here. However, martensite might appear should stresses beyond 1400 g.mm be applied.

Clinical significance and discussion

Burstone (1981) pointed out that the modulus of elasticity should be used to predict the amount of moment delivered by an orthodontic wire, each alloy having a characteristic modulus of elasticity. Their linear regression lines for the unloading curve, as observed experimentally, should approximate the moment conditions during clinical use.

The same author, in 1985, limited this statement to conventional wires, the newer wires made of alloys containing nickel–titanium components behaving differently: their unloading is complex and their stiffness depends upon the amount of activation. Indeed, the elasticity modulus varies according to the amount of activation. Every actual force system should therefore be established experimentally.

The series of tests described in this investigation suggest that further studies of the properties of Ni–Ti wires are required. A previously overlooked factor, environmental temperature, appears to be critical. Intra-oral temperatures are determinant in the amount of restoring torque delivered by such wires, and their moments should be measured under the same conditions.

The specific and constant behaviour of standard orthodontic wires made of conventional alloys is a widely established fact, within the range of body temperatures. When applied stress remains below their elastic limits, the strains remain proportionate to the amounts of stress. Furthermore, when stresses are relieved from the wires, they return to their former shapes.

Under similar conditions of clinical use, this investigation shows that the elastic modulus of Ni–Ti wires varies.

Ni–Ti and Copper Ni–Ti alloys are used to induce tooth movements with light forces over long distances. It might very well be that stresses measured experimentally at room temperature develop a greater amount of stress than expected once they are active at the higher temperature currently measured in the mouth.

This is why tests conducted at room temperature, which are acceptable for conventional orthodontic wires, are clearly insufficient to understand fully the behaviour of Ni–Ti alloys under clinical conditions of use. Additional investigation should also be made at mouth temperatures.

Data pertaining to commercially available orthodontic wires may be misleading if they have been established under unrecorded temperatures. Strict observance of unreliable data might lead to the application of much higher moments than would be clinically safe.

Conclusion

True knowledge of the magnitude of torque applied to the teeth for a given amount of torsion of orthodontic wires is of prime importance to the clinician, and this study shows that the manner in which Ni–Ti and Copper Ni–Ti alloys deliver moments during loading and unloading is closely related to temperature.

Further tests are planed to bring experimental conditions even closer to the clinical environment, loading edgewise wires at room temperature (22°C) and unloading them at mouth temperature (37°C).

Orthodontists have long varied the forces used to move the teeth by changing the thickness of wires made from the same alloy. Later, new alloys became available, which could be used to modulate forces created by successive wires of identical cross-section.

The fact that the nature and magnitude of forces applied to the teeth depend on the transformation temperatures within the wires and the close relationship with buccal temperature should be emphasized.

This investigation suggests that, in the future, orthodontists will rely on wires having different elastic properties due to their various crystallographic phases.

Whenever the transformation temperature R-phase → austenite (Rf′) or martensite → austenite (Af) is lower than the buccal temperature, almost constant torques are delivered to the teeth. This was observed with US Ni–Ti, Neo Sentalloy F100 and Copper Ni–Ti 35.

If the transformation temperature is higher than the buccal temperatures, it implies that torques applied are intermittent, as with Copper Ni–Ti 40.

Moreover, every time the transformation temperature Rs or Ms of the alloy used is lower or equal to the buccal temperature, the orthodontist can take advantage of the superelastic effect in the wires to tie them easily into the bracket without risk of bonding failure.

As a rule, the stiffness of Ni–Ti and Copper Ni–Ti wires is highly suitable when low stiffness is required and large deflections are needed. And the stiffness of Ni–Ti and Copper Ni–Ti wires is determined by the temperature.

An unexpected application of this property, when such wires are used clinically, is that uncomfortable pressure on the teeth, as experienced by patients in the initial phase following insertion of new arches, can be relieved by a simple glass of cool water, which immediately decreases the stiffness of the Ni–Ti and Copper Ni–Ti wire applied to the teeth.

A hot drink will have the reverse effect and increase the wire stiffness, and therefore make the overall appliance more effective.

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