Comparison between the mechanical behaviour of Ti-Ni and Ti-Ni-Cu shape memory alloys used for orthodontic applications

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ANNOTATION

NiTi and NiTiCu orthodontic wires with different chemical compositions, were heated to 900°C for 10 minutes followed by water quenching at 20°C. The transformation temperatures were determined by means of a flow calorimeter and the tension tests at different temperatures were carried out in order to determine the effect of the Copper on the martensitic transformation characteristics. Small chemical composition changes produce large variations in the transformation temperatures for NiTi alloys. However, for NiTiCu alloys (Cu content ranges usually between 5 and 10%) the transformation temperatures are much more stable in relation with changes in the chemical composition. A relevant result obtained is that NiTiCu alloys show stable superelasticity characteristics when cyclically loaded with small stress-hysteresis at variance with NiTi alloy. This effect is interpreted in terms of the lower rate of crystalline defects generation upon cycling in the NiTiCu alloy. It is assumed that the optimal tooth movement is achieved when the applied force is low, stable and continuous, in order to minimise tissue destruction and to produce a constant stress on the periodontal ligament during tooth movement. The present results seem to provide a material where the control of the applied forces by the orthodontist can be successfully achieved.

Keywords: NiTi, superelasticity, shape memory effect, Orthodontics

1. Introduction

When Shape Memory Alloys are thought as candidates to be applied in medical devices, they must be able to fulfil functional requirements related not only with their mechanical reliability but also with their chemical reliability (*in vivo* degradation, decomposition and dissolution, corrosion, etc) and their biological reliability (biocompatibility, citoxicity, carcinogenicity, antithrombogenicity, antigenicity, etc). NiTi is the most important shape memory alloy used as a biomaterial, that is to say, a material that comes into contact with and is tolerated by the living tissues, without any deterioration in its properties and in its specific function after implantation. However, besides internal applications, Cu-based shape memory alloys can be used in external biomedical devices where no biocompatibility requirements need to be fulfilled (1-2).

Tooth movement during orthodontic therapy is achieved by applying forces to teeth which result in bone remodelling processes. The elastic deformation of an orthodontic wire and the subsequent release of its elastic energy over a period of time gives rise to the correcting forces. It is generally assumed that the optimal tooth movement is achieved by applying forces that are low in magnitude and continuous in nature. Such forces minimize tissue destruction and produce a relatively constant stress in the periodontal ligament during tooth movement. The pseudoelasticity of NiTi archwires allows the orthodontist to apply an almost continuous light force with larger activations that results in the reduction of tissue trauma and the patient discomfort, thus facilitating enhanced tooth movement.

In contrast, forces that are high in magnitude encourage hyalinization of the periodontal ligament and may cause irreversible tissue damage such as root resorption. The NiTi archwires produce teeth movement with greater efficiency and in a shorter time when compared to other orthodontic alloys and it is specially adequate in situations requiring large deflections of an orthodontic archwire such as preliminary bracket alignment stage in load deflection stage in orthodontic therapy (3-10).

The stress-strain curve for a pseudoelastic material shows that the stress induced Martensite → parent phase transformation, upon unloading takes place at a fairly constant stress over a significant range of wire activation with a desirable biological response (11-12).

2. Materials and Methods

NiTi and NiTiCu orthodontic archwires with different chemical compositions (Table I and II, respectively) were studied. Five samples for each alloy and for each chemical composition, with 45 mm in length and 0.457 mm in diameter, were heated to 900°C for 10 minutes and quenched in water at 20°C, resulting in an austenitic phase at room temperature. The transformation temperatures were measured by means of a calorimeter Melcor S 10. The calorimetric system used was based on a flow calorimeter which measured differential signals (ΔT) by means of thermobatteries. Temperature was measured by means of a standard Pt-100 probe. All signals were digitized through a multichannel recorder and linked to a microcomputer. M_s and A_s transformation temperatures occur when there is a sudden increment in calorimetric signal. In the same way, the final temperatures, M_f and A_f, are determined as when the calorimetric signal returns to the base line. The transformation temperatures were measured during the first heating and cooling cycle after heat treatments.

TABLE I. Chemical composition in weight percentage of NiTi wires.

Alloy	% Ni	% Ti
1	55.8 ± 0.2	44.1 ± 0.3
2	69.3 ± 0.1	30.7 ± 0.2
3	69.4 ± 0.1	30.6 ± 0.2
4	69.6 ± 0.2	30.3 ± 0.2
5	64.6 ± 0.3	36.4 ± 0.2
6	62.4 ± 0.2	37.6 ± 0.1
7	63.0 ± 0.1	37.0 ± 0.1

TABLE II. Chemical composition in weight percentage of NiTiCu wires.

Alloy	% Ni	% Ti	%Cu
1	49.0 ± 0.1	45.6 ± 0.2	5.4 ± 0.1
2	49.1 ± 0.1	45.2 ± 0.1	5.7 ± 0.2
3	49.5 ± 0.3	45.0 ± 0.1	5.5 ± 0.1
4	49.6 ± 0.1	45.0 ± 0.2	5.4 ± 0.2
5	49.9 ± 0.2	45.1 ± 0.2	5.0 ± 0.1

a) Effect of load cycling.

Fatigue tests were carried out on an servohydraulic testing machine (MTS-Bionix 858), working at a cross-bar speed of 10mm/min. The NiTi and NiTiCu specimens tested were cylinders of 0.457 mm in diameter and of 45 mm in height. The gauge length of the specimens or the distance between the grips was 37 mm. Different number of fatigue cycles were applied up to a 3% of maximum strain controlled by means of extensometry with a R=0 and 1 Hz. The stress-strain curves were used to obtain the residual deformation values and the critical stress required to induce the martensitic transformation and their variations with the number of cycles. The transformation temperatures were determined after different number of cycles.

3. Results And Discussion

The addition of even small concentrations of many third elements to Ni-Ti results in a large change in the M_s temperature such that controllable adjustments of M_s are not easily achieved (13-16). In contrast, substitution of even large concentrations of Cu does not change the M_s temperature significantly. Tables III and IV show the transformation temperatures and thermal hysteresis for different Ni-Ti and Ni-Ti-Cu shape memory alloys of different chemical compositions.

TABLE III Transformation temperatures and thermal hysteresis (°C) for the Ni-Ti Orthodontic wires studied.

Alloy	M_s	M_f	A_s	A_f	ΔT_0
1	27.2 ± 0.3	16.1 ± 0.4	20.0 ± 0.1	32.3 ± 0.7	10.1
2	23.3 ± 0.2	1.2 ± 0.3	5.1 ± 0.4	28.4 ± 0.5	10.3
3	22.4 ± 0.4	14.2 ± 0.6	20.1 ± 0.3	26.5 ± 0.9	10.5
4	20.7 ± 0.1	-5.0 ± 0.4	-1.1 ± 0.2	26.1 ± 0.8	11.1
5	10.9 ± 0.2	-9.0 ± 0.3	-2.2 ± 0.2	15.2 ± 0.7	12.1
6	23.6 ± 0.3	-1.4 ± 0.3	5.1 ± 0.4	28.1 ± 0.6	10.9
7	12.4 ± 0.4	-13.4 ± 0.1	7.3 ± 0.5	16.2 ± 0.9	11.2

TABLE IV. Transformation temperatures and thermal hysteresis (°C) for the Ni-Ti-Cu Orthodontic wires studied.

Alloy	M_s	M_f	A_s	A_f	ΔT_0
1	18.7 ± 0.1	2.1 ± 0.2	14.0 ± 0.2	37.1 ± 0.6	4.5
2	17.8 ± 0.5	1.7 ± 0.3	13.8 ± 0.2	37.0 ± 0.8	3.5
3	16.2 ± 0.4	2.0 ± 0.4	13.2 ± 0.2	36.8 ± 0.8	3.7
4	17.9 ± 0.3	1.9 ± 0.6	14.3 ± 0.4	36.9 ± 0.9	3.9
5	17.6 ± 0.2	2.3 ± 0.7	14.2 ± 0.4	37.0 ± 1.0	4.4

From these results it can be noticed that small chemical composition changes produce large variations in the transformation temperatures for NiTi alloys: a variation of 0.6 % in Ni (alloy 6: 62.4 % in Ni and alloy 7: 63.0% in Ni) produces a change in the M_s temperature of 11.2°C (alloy 6: M_s= 23.6°C and alloy 7: M_s= 12.4°C). However, for NiTiCu alloys (Cu content ranges usually between 5% and 10%) the transformation temperatures are much more stable in relation with changes in the chemical composition. In this case, a variation of 0.6 % in Ni (alloy 1: 49.0% in Ni and alloy 4: 49.6% in Ni) produces a change in the M_s temperature of 0.8°C (alloy 1: M_s= 18.7°C and alloy 4: M_s= 17.9°C). Moreover, although M_s is insensitive to the substitution of Ni by Cu, M_s decreases as Cu substitutes Ti. The presence of Cu also makes the M_s temperature less sensitive to variations in the Ni-Ti ratio. A lower concentration dependent M_s allows for easier production of commercial quantities of materials having a controlled M_s for thermal sensor and actuator uses. In Figure 1 can be observe the martensitic microstructures of NiTi and NiTiCu.

Figure 1a. Martensitic microstructure of the NiTi orthodontic wire.

. Figure 1b. Martensitic microstructure of the NiTiCu orthodontic wire

Calorimetric measurements indicate that alloys with copper have substantially more-narrow hysteresis than the binary alloy. Tables III and IV list the hysteresis values (ΔT_0), measured as the temperature difference between the peaks on the calorimetric curves upon heating and cooling. It can be seen that this hysteresis is reduced from around 10°C for the binary alloy to less than 4.5°C for material with copper. Further Cu additions decrease the hysteresis only slightly, with a 5.7%Cu alloy having a width of only 3.5°C.

The transformation stresses (β↔Stress Induced Martensite) are shown in Table V. A stress hysteresis is associated with the transformation, as the diferrence between the critical stresses (stress for inducing martensitic transformation due to loading and the reverse transformation upon unloading). This stress hysteresis is much narrower for Ni-Ti-Cu alloys (≅70 MPa) than for the binary (≅150 MPa). The narrower hysteresis of Ni-Ti-Cu alloys has practical importance in engineering applications requiring a fast response time on thermal cycling. Besides, the narrower stress hysteresis of Ni-Ti-Cu means that for a similar process condition, the unloading or reversion stress is higher. The stored energy density in the Ni-Ti-Cu superelastic material is correpondingly higher that of the binary alloy.

TABLE V. Critical stresses at different test temperatures.

	NiTi				NiTiCu			
Alloy	$\sigma^{\beta\rightarrow SIM}$ (MPa)		$\sigma^{SIM\rightarrow\beta}$ (MPa)		$\sigma^{\beta\rightarrow SIM}$ (MPa)		$\sigma^{SIM\rightarrow\beta}$ (MPa)	
	20°C	37°C	20°C	37°C	20°C	37°C	20°C	37°C
1	230 (15)	320 (22)	65 (9)	151 (19)	150 (17)	287 (24)	55 (5)	211 (19)
2	235 (23)	331 (29)	70 (8)	155 (24)	155 (22)	291 (34)	60 (10)	215 (23)
3	247 (18)	333 (32)	77 (7)	156 (18)	160 (32)	299 (31)	65 (32)	216 (9)
4	260 (12)	350 (20)	79 (3)	166 (18)	154 (3)	290 (21)	59 (7)	216 (17)
5	280 (12)	354 (23)	80 (5)	160 (12)	159 (6)	287 (16)	61 (12)	214 (23)

A relevant result obtained is that in contrast to what happens in NiTi alloys, NiTiCu alloys show stable superelasticity characteristics when cyclically loaded with small stress-hysteresis. On the other hand, it is clear that the hysteresis is smaller and the change of stress-strain curve by cyclic deformation is more stable in the Ni-Ti-Cu alloy, as it can be observed in Figure 2.

The stress-strain curves at different number of cycles were used to obtain the variation of the critical stress necessary to produce the superelastic effect, and to determine the optimum working stress for the alloy. Figure 3 shows the critical stress required to induce the martensitic transformation as a function of the number of cycles. From the results shown in Figure 3 it can be noticed that the critical stress required to induce the martensitic transformation decreases with the number of cycles up to saturation for the NiTi whilst the Ni-Ti-Cu does not present any changes.

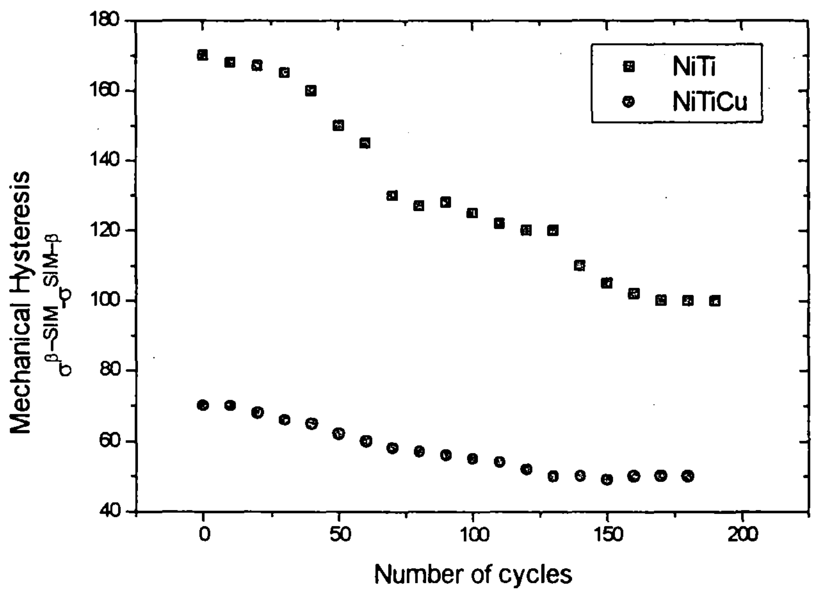


Figure 2. Mechanical hysteresis versus the number of load cycles.

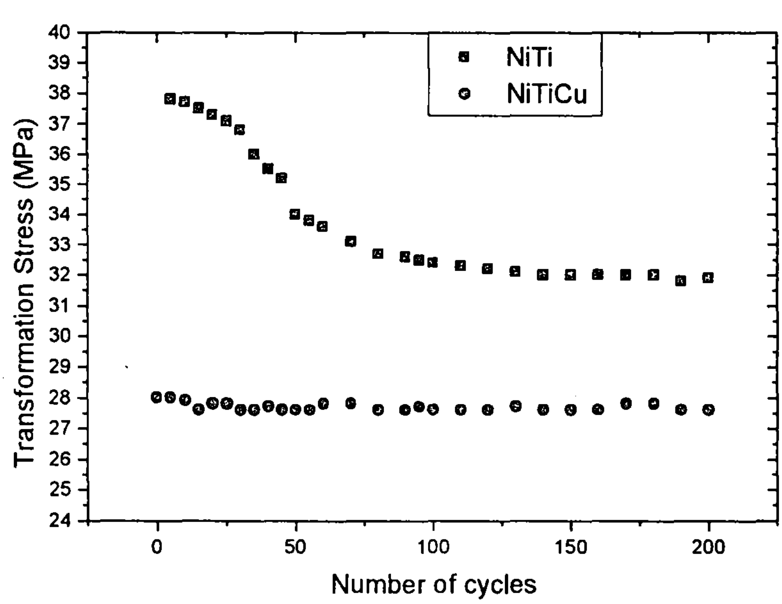


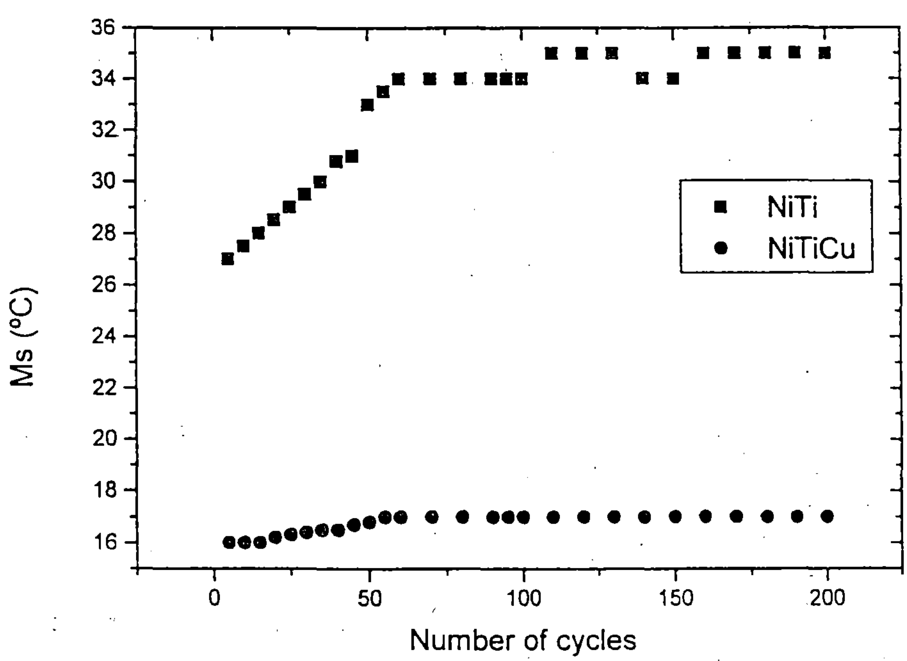
Figure 3. Transformation stress versus the number of cycles for NiTi and NiTiCu shape memory alloys.

The above results of the small stress-hysteresis and the stability against cyclic loading suggest that the interface between the parent and martensitic phases can move easily without producing a large amount of defects such as dislocations. This is reasonable by considering the fact that the habit plane of the Ni-Ti-Cu alloy is plain, since the lattice invariant shear is not necessary in order to form the habit plane in this alloy whose crystal structure is orthorhombic (1).

It should be emphasized that above a certain number of cycles, about 50 load/unload cycles in this case, the critical stress for NiTi alloys reaches saturation (13). This suggests that the transformation is stabilized around a practically constant stress and, even more important, this occurs at a relatively low number of cycles. The results of the transformation temperatures in relation to the load cycles are shown in Figure 5. It was observed that the transformation temperature to martensite, M_s, increases whilst the transformation stress decreases with the number of cycles. Such decrease in the transformation stress may be understood by the arrestment of martensitic plates by dislocations, inducing the nucleation of new plates at M_s and producing an increase of the transformation temperature.

The increase in the A_s temperature is also shown; the reason for this is that the sample contains a large amount of stabilized martensite when the load cycles increase. The M_f and A_f are practically constant in relation to the number of cycles. For the NiTiCu, the transformation temperatures are kept constant with the load cycles. The above results of the small stress-hysteresis and the stability against cyclic loading suggest that the interface between the parent and martensitic phases can move easily without producing a large amount of defects such as dislocations. This is reasonable by considering the fact that the habit plane of the Ni-Ti-Cu alloy is plain, since the lattice invariant shear is not necessary in order to form the habit plane in this alloy whose crystal structure is orthorhombic (1).

It is assumed that the optimal tooth movement is achieved when the applied force is low, stable and continous, in order to minimize tissue destruction and to produce a constant stress on the periodontal ligament during tooth movement. The present results seem to provide a material where the control of the applied forces by the orthodontist can be successfully achieved.



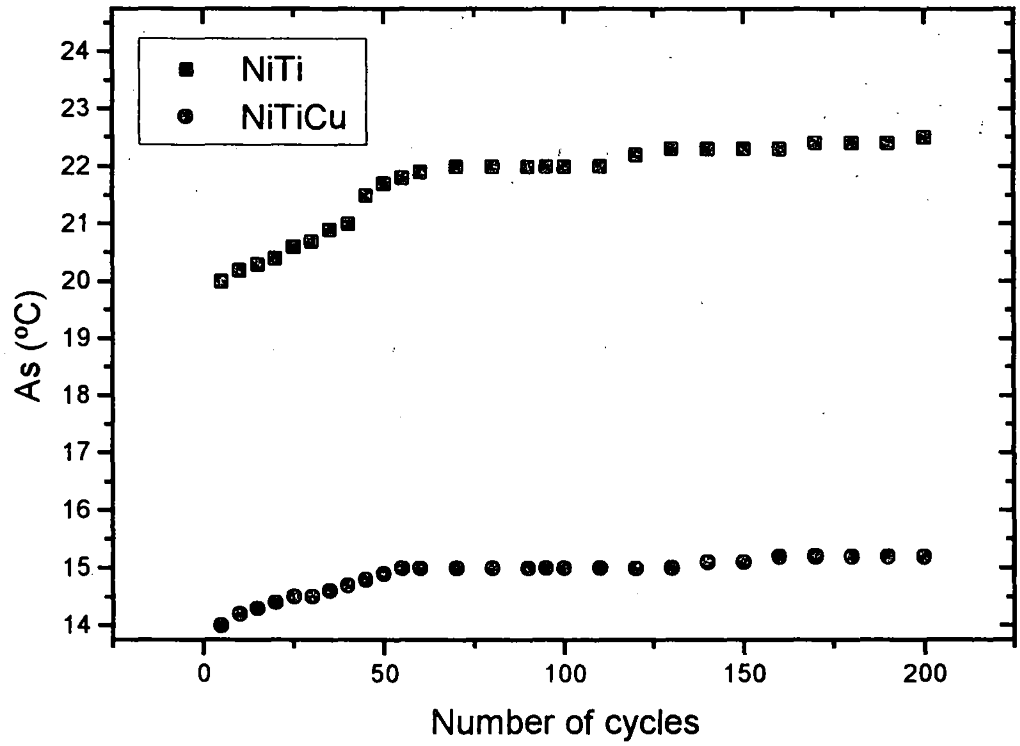


Figure 4. Transformation temperatures versus the number of cycles.

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