Journal of Multidisciplinary Engineering Science and Technology (JMEST)
ISSN: 2458-9403
Vol. 8 Issue 2, February - 2021

Characterization of the thermomechanical behaviour in correlation with structural aspects of Ni-Ti orthodontic arch wires

Elena Brandaleze

Process Technology Group: Metallurgy Department - DEYTEMA Center, Universidad Tecnológica Nacional Facultad Regional San Nicolás, San Nicolás, Argentina, jserbali@frsn.utn.edu.ar

María Agustina Reano

Process Technology Group: Metallurgy Department - DEYTEMA Center, Universidad Tecnológica Nacional Facultad Regional San Nicolás, San Nicolás, Argentina, areano@frsn.utn.edu.ar

Joel Serballi

Process Technology Group: Metallurgy Department - DEYTEMA Center, Universidad Tecnológica Nacional Facultad Regional San Nicolás, San Nicolás, Argentina, jserbali@frsn.utn.edu.ar

Vanina Tartalini

CCT Rosario – CONICET, Esmeralda y Ocampo, 2000, Rosario, Santa Fe, Argentina tartalini@ifir-conicet.gov.ar

Carolina García Díaz

Process Technology Group: Metallurgy Department – DEYTEMA Center, Universidad Tecnológica Nacional, Facultad Regional San Nicolás, cgarciadiaz@frsn.utn.edu.ar

Pablo Risso

CCT Rosario – CONICET, Esmeralda y Ocampo, 2000, Rosario, Santa Fe, Argentina risso@ifir-conicet.gov.ar

Martina Ávalos

IFIR – CONICET, Esmeralda y Ocampo, 2000, Rosario, Santa Fe, Argentina avalos@ifir-conicet.gov.ar

***Abstract*— In this paper, results of a study on commercial orthodontic arch wires (with square cross section) are presented. The samples selected include two conditions, before and after use. One of the main objectives of the paper considers the thermomechanical behaviour of the NiTi wire determined by a combination of differential scanning calorimetry (DSC) and measurements obtained through a specific experimental test developed for this study. The phase transformations in relation with temperature are also discussed. The thermodynamic stability of NiTi wire phases are predicted by simulation applying FACT SAGE 8.0 and corroborated with the structural information obtained by X Ray diffraction and observations by microscopy techniques. As it is known, clinical application requires a combination of shape memory effect at transition temperature range (TTR), superelasticity (that promotes tooth movement), suitable corrosion resistance, surface roughness characteristics, mechanical properties and biocompatibility. This paper presents results of surface roughness characteristics studied on wires in two conditions, before and after use. In addition, results of the thermomechanical behaviour of NiTi wires obtained by experimental tests at odontological treatment conditions are discussed. The transformation temperatures (TTR) of the samples were determined by differential scanning calorimetry (DSC). The information obtained is correlated with results of a thermodynamic simulation and microstructural aspects of the NiTi alloys systems selected for this study.**

Keywords: shape memory effect; thermomechanical behaviour; arch wires; orthodontic treatment; phase transformation temperature

I. INTRODUCTION

NiTi alloys are considered one of the most relevant material systems that present shape memory behaviour. These types of alloys, after an apparent plastic deformation recovers the original shape by heating. The shape memory behaviour associated with elastic deformation is named superelasticity. Both effects are due to the thermoelastic martensitic transformation. For this reason, NiTi alloys are applied to generate force/ movement (by shape memory effect) and to accumulate energy (due to superelasticity phenomenon).

Some researchers provide very useful information on NiTi alloys wires behaviour which helps in the selection and application of the appropriate arch wire according with the specific orthodontic treatment [1, 2]. The arch wire selection allows to predict treatment results, considering a minimal patient malaise. The load caused by orthodontic wires determine the design of the treatment program [3]. In addition, it is relevant to consider the knowledge on the biomechanical and clinical applications of orthodontic wires.

Orthodontics works by an intentional force application, in order to promote the transduction phenomenon (the transformation of a physical force into a biological response) that executes the guide and control of tooth movement. Different biological responses depend on the force term/duration and its magnitude. W. Wichai et al. in [4], mentioned that orthodontic wires should be able to move teeth with a light continuous force.

The force applied to the tooth is derived through the periodontal ligament, generating a stress situation at the tissue that induces the bone remodeling. This effect involves: bone resorption on the pressure side and bone apposition on the tension side. This means maintenance of the periodontal ligament structure and thickness. In consequence, the dental piece and the attachment apparatus move together. The forces considered as therapeutic forces are those classified as second grade forces: that is lower than capillary blood pressure (20-26 g/cm^2), allowing the periodontal ligament to preserve the metabolic viability without causing root resorption [3].

A dynamic friction force can also affect the orthodontic treatment. The friction force is determined by the surface roughness of the wires. Roughness influences the interaction of the wire with its environment. Friction is considered the resistance force between surfaces with opposed motion, between orthodontic appliances parts [5]. The topography of orthodontic arch wire affects the sliding mechanics and the friction in contact with the bracket [4, 5]. Rudge et al in [5] also informs the relevance of other factors apart from friction force such as corrosion potential and plaque accumulation that could induce caries and gingivitis risk. For this reason, in this study the roughness surface characteristics of both selected wires conditions were also studied (before and after use).

Different types of arch wires with different alloys and cross sections (round, square, rectangular, etc.), are used in orthodontic appliances during all stages of orthodontic treatment. It is relevant to consider that NiTi alloys behaviour presents a high sensitivity associated with chemical composition [1]. For this reason, it is very important to dispose a deep information on the phases stabilities, transformation temperatures, knowledge on the microstructure and mechanical properties evolution of the arch type to be use. In this paper a wire of NiTi alloy with a square section is characterized. The shape memory effect results from the superelasticity and thermoelastic behaviour and constitutes the main characteristic of thermally activated NiTi arch wires. This behaviour is closely related to the transition temperature range (TTR). In this case, the thermal behaviour and the phase transformation temperatures range (TTR) were determined applying differential scanning calorimeter (DSC) in coincidence with different authors [1, 6-8]. The mechanical behaviour in relation to temperature was studied by a test performed applying a specific equipment developed for this study. The test was carried out at temperatures between 0°C to 100°C, in order to cover the oral temperature intervals.

The surface roughness and microstructure of the samples were studied applying different microscopy techniques such as: optical microscopy (including differential interference contrast, DIC), atom force microscopy (AFM) and scanning electron microscopy (SEM with EDS analysis). The crystalline phases identification was carried out by micro X rays.

The stability and evolution of the phases in relation to temperature were predicted by a thermodynamic simulation applying the software FACT SAGE 8.0, using an analogous methodology as described in [9-11].

II. METHODOLOGY

Two NiTi arch wires identified as A (before used condition) and B_{pu} (after used condition) were selected for the study. The characteristics of them are detailed in Table I.

TABLE I. WIRE SAMPLE CHARACTERISTICS

Sample	Type	Transversal section	Size
A	Superelastic	Square	0.16”x 0.16”
B_{pu}	Superelastic	Square	0.16”x 0.16”

In order to evaluate the surface roughness aspect of the wires before and after use the samples were observed applying a stereoscopic magnifying glass Olympus ZL71, optical microscopy by differential

interference contrast (DIC), atom force microscopy by a Core AFM Nanosurf instrument and scanning electron microscopy (SEM) FEI Quanta 200 equipped with an EDAX – EDS analysis. The microstructural aspects of the wires were observed in transversal view samples prepared with cold curing resin, polished with SiC (up to 1200 µm) and diamond paste (up to 1 µm) and etched with a metallographic solution. The microstructure was observed by an optical microscope OLYMPUS GX 51 (with image analyzer system Material Plus) including bright field and DIC. The structural study was completed by scanning electron microscopy (SEM+EDS) using a FEI Quanta 200 microscope. In addition, microhardness measurements were performed applying a LECO LMT 300 instrument. The crystalline phases were identified with micro X rays carried out with a X´ Panalytical Empyrean – USP diffractometer and the software Match! 3.

The thermomechanical behaviours evolution associated with temperature was established through a test methodology and procedure (applying an equipment developed for this purpose) at a temperature range between 0°C to 100°C. The main objective is to stablish the arch wire shape variation with temperature. The description of the measurement procedure steps is:

- During the test the arch wire is heated up to 100°C onto a gridded plate and the temperature is continuously measured by a laser gun.
- The experiment is registered on video with a camera placed on the top of the equipment, in order to get a view of the top arch.
- The deformation measurements procedure is carried out on digitalized images (captured from the video), applying the image analysis system TSView 7. The selected images include different t(s) and T(°C) conditions.
- At each image, 6 longitudinal measurements in the “L” orientation and 7 in “T” orientation were performed. All of them coincident with the gridded of the plate, see Fig 1.
- The measurements obtained in all of the considered conditions are compared with the values corresponding to the image captured at time t(0) and room temperature (as reference).

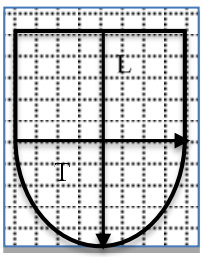


Fig 1. Scheme of the measurements performed at the orientations L and T on the arch wire image.

The transformation temperature ranges (TTR) were determined by differential scanning calorimetry (DSC) applying a Shimadzu DTG 60H instrument. The test was carried out considering a heating and cooling rate of 10°C/min. The phases evolution and stabilities were predicted by a thermodynamic simulation performed by the software FactSage 8.0. This tool provides information about phase transformations and precipitation phenomena that can occur at different temperature conditions. In this case, the Equilib module was applied, considering the data bases: FTmisc, FTOxCN and FactPS. The temperature range of the simulation was 0°C to 100°C.

III. RESULTS AND DISCUSSIONS

A. *Arch wires chemical composition*

In this type of NiTi arch wires, it is relevant to consider the chemical composition due to the impact produced on the properties and making process. The chemical composition of the samples A and B_{pu} are detailed in Table II.

TABLE II. WIRE SAMPLES CHEMICAL COMPOSITION

Wire	% Ni	%Ti	%Fe	%Cu
A=B_{pu}	57.84	rest	0.09	< 0.01

B. *Wires roughness surface characterization*

Surface characteristics of the preformed NiTi arch wires may affect the sliding mechanisms, because they determine the friction coefficient between the arch wire and the bracket. It is important to consider that the friction depends on the absolute roughness of both individual surfaces [4, 5].

The surface roughness of the arch wire A was observed applying different microscopy techniques such as stereoscopic magnifying glass and differential DIC. The surface aspect of sample A obtained by DIC is presented in Fig. 2. Numerous longitudinal and parallel striations are identified. All of them are aligned along the wire´s longitudinal axe. The striations characteristics were also observed by scanning electron microscopy (SEM), see Fig 3.

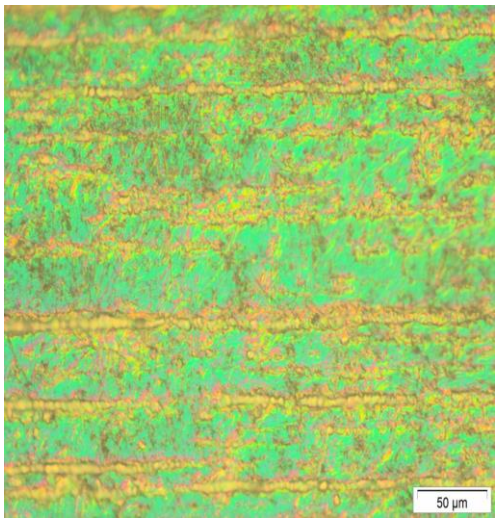


Fig.2. Roughness aspect observed on sample A.

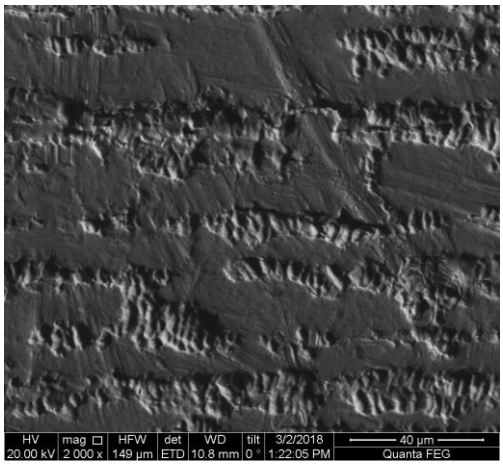


Fig. 3. Surface roughness of wire A observed by SEM.

Sample A surface profile was also studied applying an atomic force microscopy (AFM) scanning on different zones of the transversal surface to evaluate the depth of striations. The results allow to verify that the striations average depth value is ~ 0.27 µm, σ: 0.02. This value is consistent with the reported for different orthodontic arch wires in [4].

In the case of wire B_{pu}, the aspect of the surface is shown in Fig 4. Different types of particles and crystals are embedded or adhered in the striations as it is observed.

The particles and crystals presence in the striations of B_{pu} sample demonstrates that the roughness not only is relevant in relation with the friction coefficient required for the orthodontic treatment. It also represents an important factor associated with problems such as: potential corrosion and plaque accumulation. The latter could increase the patient´s risk of caries and gingivitis [5].

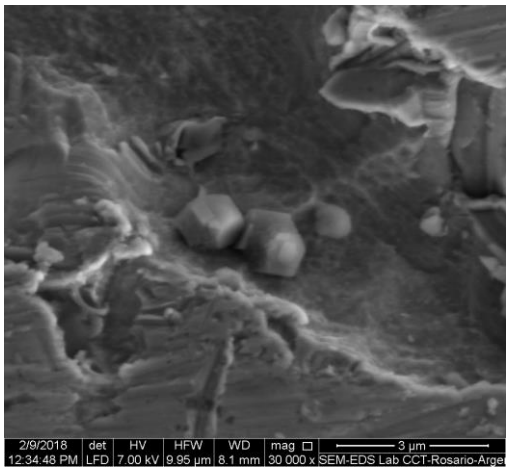


Fig.4. Roughness aspect observed on sample B_{pu} with crystals and particles presence.

The polyhedral crystals were analyzed by EDS (SEM) and present mainly content of Ca. In addition, they present low contents of Si, Al and Mg. Some particles embedded in the surface of the arch wire contains Si and Al. The majority of the particles are associated with tartar and saliva, diet and bacterial products. In addition, spherical particles of alumina were identified on the surface as a waste of toothpaste. Fig. 5

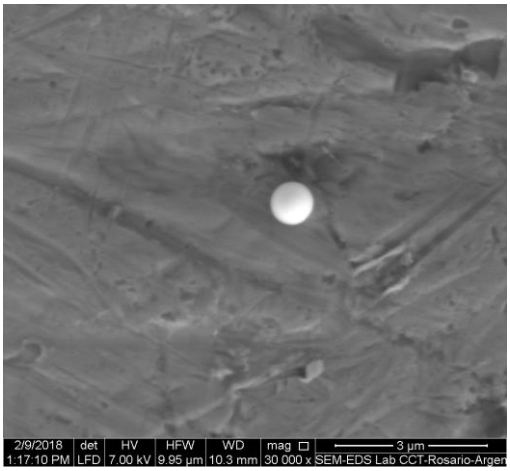
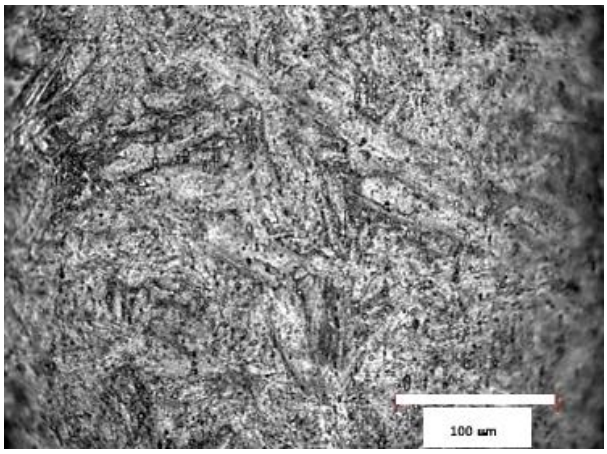


Fig.5. Spherical particles of alumina identified on B_{pu} sample surface.

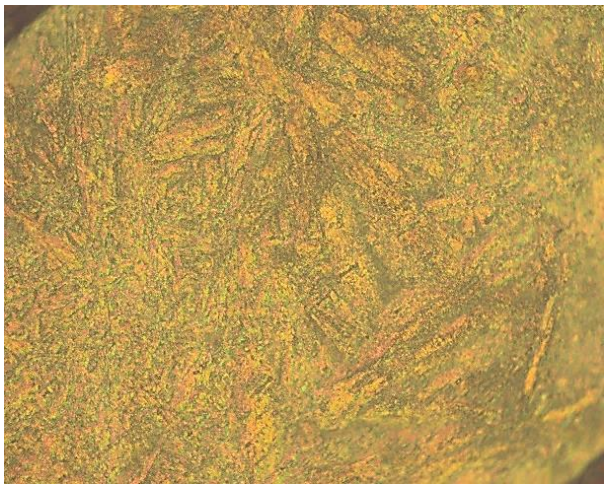
C. Microstructural characterization.

The microstructural characterization of the arch wire samples was carried out by different microscopy techniques.

The optical microscopy observations include bright field and DIC. It was possible to observe that the wire A presents a structure fully martensitic at room temperature, as it is showed in Fig 6 (a) and (b).



(a) Fully martensitic structure observed in A wire sample with light microscopy.



(b) Martensitic structure aspect observed in A wire sample with DIC microscopy.

Fig 6. Microstructural characteristics of the sample A observed by optical microscopy.

Through bright field microscopy it was possible to note the presence of precipitates particles distributed in the structure, mainly in the interface of the acicular structure. Both techniques of optical microscopy corroborate the presence of acicular structure characterized by a random orientation. The microhardness measurements were carried out applying a load of 50 mg. The wire samples present an average microhardness value of H_v= 540.

The microstructural study was completed by scanning electron microscopy (SEM) observations, including EDS analysis to determine the precipitates chemical composition. In Fig 7, the aspect of the microstructure of the sample A that presents in more detail the distribution of the precipitates in the interfaces of the acicular martensitic structure is presented. The precipitates were analyzed by EDS and it was possible to establish that they are constituted by 79,6 %Wt. Ti - 20,4 %Wt. de Ni. The detail of the precipitates´ particles is observed in Fig 8.

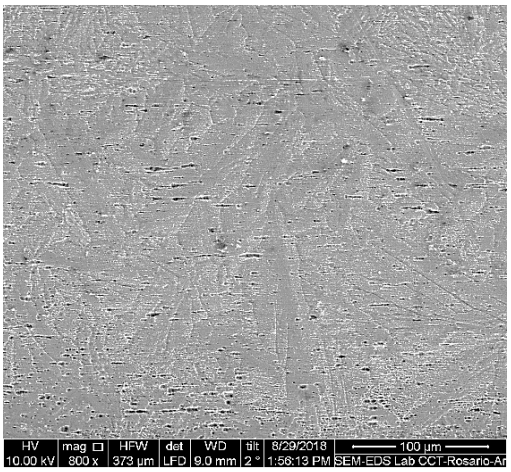


Fig 7. Microstructural characteristics of the sample A observed by SEM.

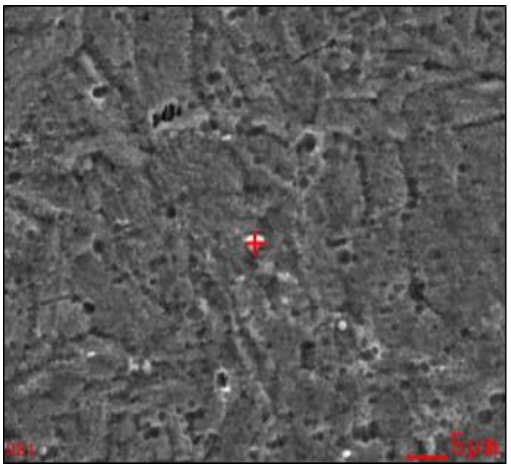


Fig 8. Precipitates present in A wire microstructure observed and analyzed by SEM with EDS.

This type of NiTi alloys presents different possibilities of precipitates formation in relation with the chemical composition and the heat treatment conditions used during the making process. The volume fraction, size and distribution of them in the structure affect the mechanical properties, the shape memory effect and the pseudoelastic behaviour. Grain boundaries promote the precipitation phenomena through the highest energy accumulated.

D. Phases identification by micro XRD.

The phases present in the arch wires were identified through micro X ray diffraction. The difractogram obtained is observed in Fig 9.

The main crystalline phases identified in the sample A are: a) $Cu_{0.5}$ $Ni_{0.5}$Ti and b) NiTi.

The $Cu_{0.5}$ $Ni_{0.5}$Ti phase with orthorhombic structure constitutes the martensitic phase “M”. The results corroborate that this solid solution constitutes the main phase present in the arch wire. In addition, a very low content of a crystalline phase of NiTi with a monoclinic structure is detected.

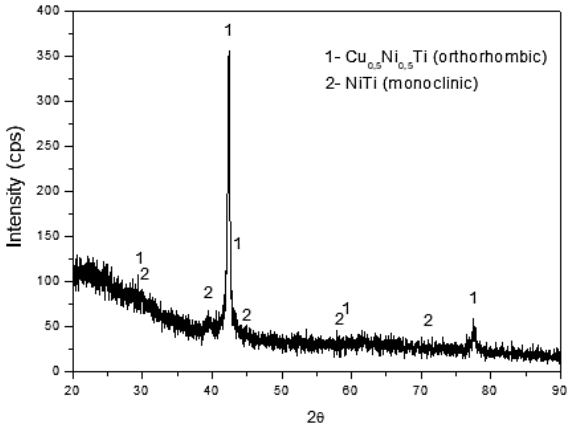


Fig 9. Crystalline phases identified in the wire samples by micro XRD.

E. Determination of TTR temperatures, deformation and thermomechanical behaviour of sample A.

The transformation temperatures range TTR of the arch wire A, are determined by a DSC test carried out considering heating and cooling at temperatures between 0°C to 100°C (that includes the oral temperature range). Endothermic and exothermic processes on heating and cooling were evident for this arch wire A of CuNiTi. On the base of the curves obtained the transformation temperatures were determined. In Fig 10 it is possible to observe TTR temperature values determined and plotted at both conditions.

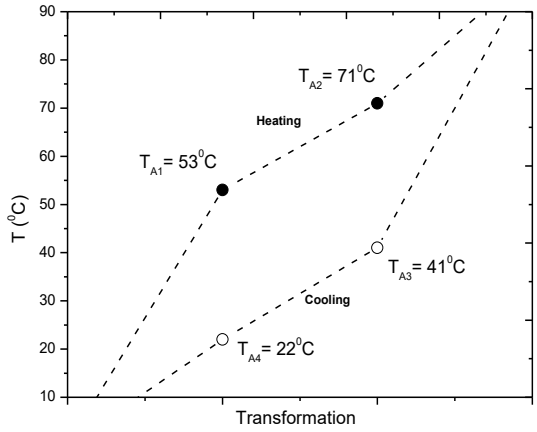


Fig 10. TTR temperature values of sample A determined by DSC at heating and cooling conditions.

The initial and final transformation temperatures of the austenite (A) and martensite (M) phases are presented in table III.

TABLE III. INITIAL AND FINAL TEMPERATURE TRANSFORMATION OF THE SAMPLE A

SAMPLE	A_S	A_F	M_S	M_F
A	53°C	71°C	41°C	22°C

During heating, the austenite phase starts to form at A_S=53°C and finishes at A_f=71°C. Between these temperatures the structure of the arch wire is biphasic (M+A). At temperatures above A_f the structure remains fully austenitic with a cubic crystalline structure and pseudoelasticaly behaviour [12, 13].

At cooling conditions, the martensite phase starts to form at M_S=41°C and finishes at M_f = 22°C. Between both temperatures the structure is also biphasic (A+M). At temperatures lower than 22°C the microstructure is fully martensitic. The martensitic transformation is a diffusionless reversible solid state phase transformation, thermally induced [14].

It is possible to consider that the presence of martensite phase in the structure at T< 41°C, progressively increases and, in consequence, the superelastic behaviour of the arch wire. The obtained results allow to establish that the most efficient range for the orthodontic treatment of the arch wire A is at T≦ 41°C (coincident with common oral temperature range). The superelastic state promotes to develop great deformation with a quasi-constant load, achieving a good response from the bone tissue during the initial period of the treatment.

The arch wire deformation associated with temperature was determined by a test carried out with an equipment and procedure developed with this purpose.

It was possible to note that during heating (at M → A transformation) the arch wire did not present an appreciable shape variation. However, during cooling at M_S (41°C), displacements of 3.5 mm in both orientation (L and T) were registered, showing a 2D effect associated to square section arch wires. These dimensions remain up to room temperature. The displacements result is consistent with the values informed in [15].

In Fig 11, the plot of the deformation evolution with temperature obtained only in L orientation, is presented. It is possible to note that the average L value during cooling, when the arch wire presents austenite phase in the structure is around ~ 58.5 mm σ: 0.12. However, at 41°C when the martensitic transformation starts, a variation of ΔL= 3.5 mm is determined. The result is justified by the volumetric variation caused due to the martensitic phase transformation that presents a displacive nature.

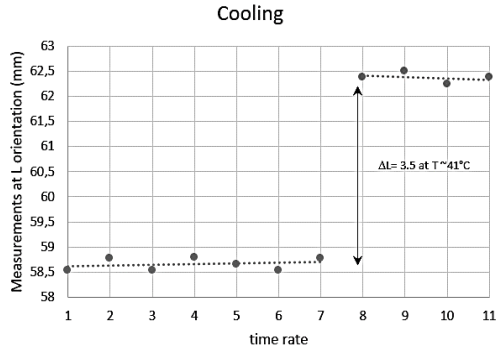


Fig 11. Average L values on sample A at different images, different times and temperatures.

F. Arch Wire phases stability predicted by thermodynamic simulation.

The Ni-Ti system is considered a binary alloy, constituted by an equiatomic intermetallic compound of Ni and Ti. In Fig 12, the phase diagram of the system obtained by Fact Sage, is observed. This intermetallic compound (NiTi), placed in the central stability field of the diagram presents a good solubility of different elements that modify the mechanical properties, the thermal behaviour and the shape memory aptitude of the arch wires. In the arch wire A, Cu is the element that is added to the alloy.

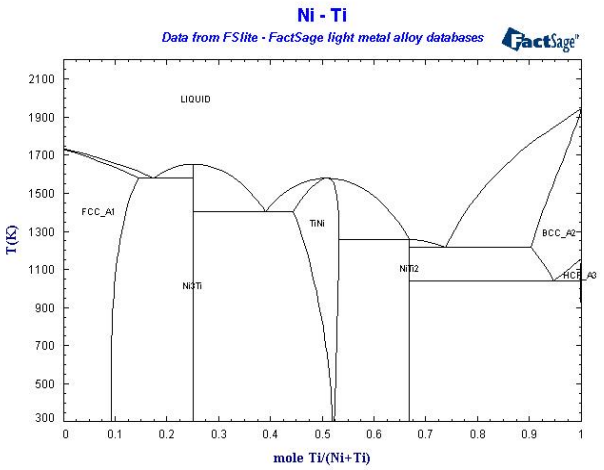


Fig 12. NiTi alloy diagram obtained by FACT SAGE.

The thermodynamic simulation carried out by FACT SAGE 8.0 involves a wide range of temperatures (-50ºC a 500ºC) in order to verify the possible phase transformations of the wire A and their stability conditions. The predictions consider a comparison between the system of the sample A and a NiTi reference system (without alloy elements). The results show a good thermodynamic stability of the wire A alloy, through the lower Gibbs free energy values in relation with the reference system NiTi considered (ΔG_{sys}), at all temperatures (see Fig 13). It is also evident that the alloy sensitivity varies with the chemical composition. In this case, the effect of Cu addition increases the NiTi phase stability.

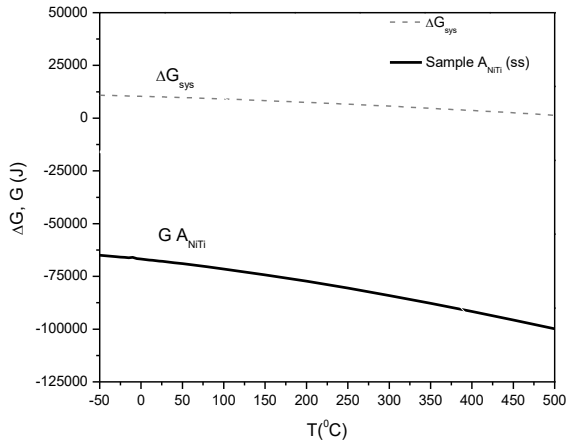


Fig 13. Comparison between the free energy values of the wire A alloy (A_{NiTi}) with respect to the reference NiTi system at different temperatures.

The thermodynamic simulation predicts that the possible stable crystalline phases of the alloy A at oral temperatures range are: 88% NiTi (ss) with solubilized Cu, low contents of $NiTi_3$-$TiNi_3$ 12%, traces of Fe_2Ti. At temperatures > 300°C (during the making process) a crystalline phase fcc constituted by 99% of Cu and traces of Fe and Ni also could be present in the structure. This information is useful for the industry because this type of wires is annealed at temperatures ~ 350°C in order to adjust the TTR values required.

The correlation of the phases predicted by FACT SAGE with the micro X ray diffraction and microscopy results allow to confirm that the main crystalline phase present in sample A (at room temperature) is the NiTi solid solution with Cu, constituting the orthorhombic phase $Cu_{0.5}Ni_{0.5}Ti$, responsible for the shape memory effect. In addition, precipitates of Ti_3Ni (in agreement with the phase diagram of Fig 12) were identified as been distributed in the grain boundaries. This type of precipitates are formed by diffusion mechanism in the grain boundaries [12-15]. In these materials, precipitates control the hardening phenomenon of the alloy [16].

The thermal behaviour obtained by DSC tests indicates that the martensitic orthorhombic phase $Cu_{0.5}Ni_{0.5}Ti$ (during heating) starts the M → A transformation at A_s= 53°C and finishes at A_f= 71°C. Above this temperature the arch wire remains in pseudoelasticaly austenite cubic phase. During cooling the phase $Cu_{0.5}Ni_{0.5}Ti$ starts the martensitic transformation at M_s=41°C and finishes at M_f=22°C. This type of transformation can progress either by a temperature variation or by the application of stress, which results in strains that induce the phase transformation. The equilibrium in this case is sustained by chemical and mechanical forces. At this state the wire presents a superelastic behaviour [14].

It is important to consider that M ↔ A transformation is reversible and due to the displacive nature, the movement of atoms is fully coordinated. The composition, the ordering of atomic structures and

the number of crystallographic lattice defects are the same. However, it is possible to consider that the transformation is crystallographically reversible (because the interfaces retain their reversibility). But it is not fully thermodynamically reversible, which in turn means that it does not proceed through thermodynamic equilibrium states. It is relevant to mention that the interfaces between martensite and austenite are coherent because the phase transition displacement field is continuous [14].

The austenite phase is considered a high symmetry cubic phase. On the contrary, the martensitic phase is a low orthorhombic (in the A sample case) symmetry phase. The differences between both phases is due to the variations of the orientation possibilities (24) of the habit planes. The martensitic phase generated during cooling presents highly mobile twins boundaries that provide a good deformation capacity.

In agreement with [17], the results obtained in this study indicate that the response of the arch wire not only depends on the chemical free energy, it also depends on the microstructural factors. Elastic energy is stored in the material through non-dissipative process during (mainly in the interface M-A). However, it is relevant to consider that it also exists the presence of energy dissipated by the friction between the arch wire and the bracket during the orthodontic treatment.

IV. Conclusions

The information obtained through the differential thermal analysis (DSC) test, the thermomechanical test developed for this study, in correlation with results of the microstructural characterization (including microscopy results and crystalline phases identification by micro XRD) and the thermodynamic study, allow to determine the transformation temperatures range (TTR) of the arch wire A and to understand the crystalline phases evolution of the alloy at different temperatures conditions.

The thermomechanical test carried out on sample A allows to determine that the transformation from austenite cubic phase to orthorhombic martensite phase produces a variation of $\Delta L = \Delta T = 3.5$ mm in the arch, as result of the martensitic displacive transformation. This means that exist 2D displacements that are common in square arch wires section. The measurements percentage relative error does not exceed the 1.6%. It is also important to consider that the arch wire is appreciably “stretched” transversely and longitudinally (2D) during cooling from M_f. Nevertheless, not variations are registered during heating when the transformation is $M \rightarrow A$. The integration of the results permits to verify that the arch wire A, during the 79% of the time at which the oral temperature is between 33°C -37°C presents a superelastic orthorhombic martensitic structure, which is the most efficient condition for the orthodontic treatment.

The characterization of the roughness surface of the arch wires also contributes with useful information to consider for the orthodontic treatment and to avoid other problems such as plaque accumulation or potential corrosion that could affect the patient. In the A and B_{pu} arch wires the striations depth is in agree with values informed in the literature.

The thermodynamic study results, integrated with the structural characterization by microscopy and micro XRD tests, make it possible to obtain an integral knowledge of the structure. It was possible to verify that the main crystalline phase that constitutes the alloy in arch wire A is $Cu_{0.5}Ni_{0.5}Ti$, which is the responsible for the shape memory effect. In addition, precipitates of Ti_3Ni are present in the grain boundaries, and contribute to the wire mechanical behaviour and hardness.

Acknowledgment

The authors wish to thank Universidad Tecnológica Nacional (Argentina) for the financial support to the research project and to Dr. Fernando Vernilli from USP, Brazil for the relevant information obtained through the micro X ray diffraction tests.

References

[1] M. Abbasi, A. Kermanpur, R. Emadi, “Effects of thermo-mechanical processing on the mechanical properties and shape recovery of nanostructured Ti50Ni45Cu5 shape memory alloy”, Procedia Materials Science, vol. 11, pp. 61-66, 2015.

[2] S. Prokoshkin, V. Brailovski, K. Inaekyan, A. Korotitskiy, A. Kreitcberg, Thermomechanical treatment of TiNi Intermetallic-based Shape memory alloys, Materials Science Foundations, vol. 81-82, pp.240-341, 2015.

[3] A. Wilchelhaus, H.F. Wolf, Orthodontic Therapy: Fundamental Treatment Concepts (Color Atlas of Dental Medice), Thieme Publishers Stuttgart, 2018.

[4] W. Wichai, R. Isarapatanapong, N. Anuwongnukroh and S. Dechkunakorn, Grain Structure and Surface Roughness of Four Commercial NiTi Orthodontic Archwires, Solid State Phenomena, vol. 266, pp 257-263, 2017.

[5] P. Rudge, M. Sherriff, D. Bister, A comparison of roughness parameters and friction coefficients of aesthetic archwires, European Journal of Orthodontics, pp. 49-55, 2015.

[6] J. Burow, E. Prokofiev, C. Somsen, J. Frenzel, R. Z. Valiev and G. Eggeler, Martensitic transformations and functional stability in ultra-fine grained NiTi shape memory alloys, Materials Science Forum, vol. 584-586, pp 852-857, 2008.

[7] O. Bartwart, J.M. Rollinger and A. Burger, An evaluation of the transition temperature range of super-elastic orthodontic NiTi springs using differential scanning calorimetry, European Journal of Orthodontics, vol. 21, pp. 497-502, 1999.

[8] T. S. Spini, F.P. Valarelli, R.H. Cançado, K.M. Salvatore de Freitas, D. J. Villarinho, Transition temperature range of thermally activated nickel-titanium archwires, J. Appl. Oral Sci., vol. 22-2, pp 109-117, 2014.

[9] A. Kostov, B. Friedrich and D. Živković, Thermodynamic calculations in alloys Ti-Al, Ti-Fe, Al-Fe and Ti-Al-Fe, Journal of Mining and Metallurgy, vol. 44 B, pp. 49 – 61, 2008.

[10] M. Romanyuk, M. Avalos, E. R. Benavidez and E. Brandaleze, Correlation between structural aspects and mechanical properties of an Interstitial Free steel for automotive application, Advanced Materials Proceedings, vol. 3, pp 408-413, 2018.

[11] A.I. Kostov, D.T. Živković, V. R. Ćosović, Thermodynamic characterization of shape memory AlNi-Fe alloys using FactSage, 15th International Research/Expert Conference ”Trends in the Development of Machinery and Associated Technology” TMT 2011, Prague, Czech Republic, pp 217- 220, 2011.

[12] G.F. Bastin, G. D. Rieck, Diffusion in the Titanium-Nickel System: I. Occurrence and Growth of the Various Intermetallic Compounds, Metalalurgicall Transaction, vol. 5, pp 1817-1826, 1974.

[13] T. Castro da Silva, D. Monteiro Rosa, E. Paulo da Silva, Effect of the Cooling Time in Annealing at 350oC on the Phase Transformation Temperatures of a Ni55Ti45 wt. Alloy, Advanced Materials Research, vol. 1120-1121, pp 958-961, 2015.

[14] S. X. D. Karamichailidou, The Unique Properties,Manufacturing Processes and Applications of Near Equatomic Ni-Ti Alloys, PhD. Thesis, University of Thessaly, Department of Mechanical Engineering Laboratory of Materials, 2016.

[15] R. De Santis, F. Dolci, A. Laino, R. Martina, L. Ambrosio and L. Nicolais, The Eulerian buckling test for orthodontic wires, European Journal of Orthodontics, 30, pp. 190-198, 2008.

[16] C. D. Cirstea, E. Karadeniz-Povoden, E. Kozeschnik, M. Lungu, P. Lang, A. Balagurov and V. Cirstea, Thermokinetic Simulation of Precipitation in NiTi Shape Memory Alloys, Materials Science and Engineering, vol 209, pp. 1-7, 2017.

[17] P. Garrec, B. Tavernier, L. Jordan, Evolution of flexural rigidity according to the cross- sectional dimension of a superelastic nickel titanium orthodontic wire, European Journal of Orthodontics, 27, pp. 402-407, 2005.