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Characterization of Phase Transformations and Chemical Composition in Rotary and Reciprocating Nickel-Titanium Endodontic Instruments

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Abstract

Background: The mechanical stresses to which the files are subjected differ depending on the type of movement. Therefore, the flexibility and resistance of the instruments must vary according to clinical needs. Which depends on the chemical composition and thermal treatment of the material, which gives it the atomic arrangement.

Objectives: Analyze the physical, chemical and thermal properties of rotary and reciprocating instruments.

Methods: The reciprocating and rotary instrumentation systems: WaveOne Gold, Reciproc Blue, Vortex Blue and V-Taper were evaluated under SEM, as well as their chemical composition by ICP-OES analysis, the phase transitions through DSC, and structurally by X-ray diffraction.

Results: Chemical analysis determined that the NiTi alloys, corresponding to Vortex Blue and V-Taper 2H, have an almost 1:1 Ni:Ti atomic ratio, but with a significant variation in the Ni content. Likewise, a low Au content was detected in the alternative WaveOne Gold system. Regarding the thermal analysis, the alternative V-taper 2H system showed a double exothermic transition associated with an intermediate rhombohedral phase known as R phase. The diffraction patterns show the transition of the austenitic phase.

Conclusions: Thermochemical analysis, Rotary Instruments, Reciproc Blue, Vortex Blue, V-Taper 2H, and WaveOne Gold.

Keywords: Nutrition, Therapy, Cancer management, Oncology

Introduction

Shape memory nickel-titanium alloy has revolutionized the treatment of curved canals. Its superior mechanical resistance and ductility impart greater safety to rotary instruments. These instruments reduce endodontist fatigue and decrease postoperative sensitivity in patients (1,2). The composition of the alloy plays an important role in the pseudoplastic properties of the material, then alloys rich in Ni (50.7%) must be used (3,4). A minimal change in the composition can give a drastic difference in the transformation temperature and interatomic ordering, affecting the mechanical behavior of the instrument. The instruments usually are subjected to different heat treatments, with the main objective of improving the flexibility and resistance to torsional fatigue since the instruments must support the forces generated during the mechanized movements inside the canal, according to the kinematics of the instrument. The inter-atomic arrangement of NiTi, generated during heat treatments, modifies the conditions in which the material begins and ends its phase transition from martensite to austenite or vice versa. The two steps via are associated with the formation of an intermediate phase called the R or rhombohedral phase. It has been reported that such R phase arises from the additional aging treatment, generated by Ti_3Ni_4 precipitates (1,5-7). Likewise, it should be noted that each of these phases provides specific properties to the material such as the flexibility conferred by the soft and ductile martensitic phase, while the austenitic phase is characterized by being more rigid. While the intermediate phase R is characterized by high resistance to fatigue. Therefore, it has special interest in the manufacturing process of endodontic instruments (1,8,9).

Regarding these phase transitions, it should be emphasized that they can be induced by an increment in temperature or by stress, being the stress transition to what the instruments are subjected during root canal treatment, which generates bending fatigue and a torsional overload on the instrument (10). Therefore, it is essential to determine the crystalline phase in which the instruments are, at the beginning of the treatment. Thus, it is possible to have information about the flexibility or rigidity of the instrument, as well as evaluate the phenomenon of the evolution of phase transitions, which will help to understand the behavior of each of the systems during the root canal treatment. That is, if the flexibility of the instrument is maintained because of the martensitic state, or if the system changes the organization of the crystals, promoting the austenitic state (10,11).

Evenly, another important factor to consider in the selection of a rotary instrument is based on its design, which considers features like the type of tip and taper design, rake angle, helical angle, and pitch since all of them play a fundamental role in the conservation and disinfection of the canal. Therefore, the manufacturing systems must go through strict quality control and comply with the ISO 3630-1:2019 test method. In this context, the purpose of this in vitro study was to make an exhaustive analysis of the design characteristics of the instruments and evaluate the physicochemical and thermal properties of commercial instruments for each of the movements. Within the single file systems, the Reciproc Blue (VDW, Munich, Germany) and WaveOne Gold (Dentsply Sirona Endodontics, York, PA, USA), are the most current versions of both brands in terms of reciprocating systems. While the Vortex Blue (Dentsply Sirona Endodontics, York, PA, USA) and V-Taper 2H (SS White Dental, New Jersey, USA)) are the version of rotary systems. These systems have been modified mainly in terms of heat treatment instead of their design, except for the cross-section of WaveOne Gold, which has been modified compared to its predecessor WaveOne. Also, a heat treatment with gold has been reported to improve the resistance to cyclic fatigue of Wave One Gold (12,13). Therefore, these treatments are responsible for providing better physical properties, especially flexibility and greater resistance to cyclic fatigue (14-16).

Materials and Methods

Materials

forty-five instruments with a length of 25mm were evaluated of the systems rotary (Vortex Blue (Dentsply Sirona Endodontics, York, PA, USA), V-Taper 2H (SS White Dental, New Jersey, USA)), and reciprocating (WaveOne Gold (Dentsply

Sirona Endodontics, York, PA, USA), and Reciproc Blue (VDW, Munich, Germany)) movements.

Thermal Characterization (Differential Scanning Calorimetry-DSC)

The enthalpy and phase transition temperatures of the rotating (Vortex Blue, V-Taper 2H) and reciprocating (WaveOne Gold and Reciproc Blue) instruments were evaluated using a DSC (TA-Instruments DSC Q200, Lukens Dr, New Castle, USA) in a range of -40 to 110°C, with a heating rate of 5°C/min under a nitrogen atmosphere. 10mg of the sample was placed in an aluminum pan. Thermogram analyzes were performed in Universal Analysis 200 software (TA instruments). Thus, the initial and final points of the martensitic transformation (Ms, Mf) and the initial and final points of the inverse transformation (As, Af) were determined.

Structural characterization (X-ray diffraction-XRD)

The X-ray diffraction technique was used to identify the crystalline structure. For this, the samples were cutted into 1cm segments and analyzed in an X-ray diffractometer (X'Pert Pro; PANalytical, Almelo, NL) with a monochromatic Cu-Kα radiation source at a wavelength of 1.54Å in the range of 40-85° 2θ with a step of 0.02° and an acceleration voltage of 35kV and current of 30mA, with an acquisition time of 12s/step. Subsequently, the X'Pert HighScore Plus software (PANalytical) was used to analyze peaks and identify crystalline phases.

Morphological Characterization (Scanning Electron Microscopy-SEM)

The morphology and design of the instruments were evaluated by scanning electron microscopy (QUANTA 200, ESEM FEI Co., Hillsboro, OR, USA). For the transversal design of the instruments, these were placed in an acrylic resin with 2% catalyst, and after 48h of setting the block 3mm from the tip of the instruments were cut with an IsoMet, Buehler™. Finally, the samples were polished with a high-speed piece (PanaMax, NSK®), starting with a rubber disc to eliminate traces of the cutting process, followed by a white felt disc, and finally with a dental table polisher with a disc flannel where the mirror finish was given for analysis.

Chemical Composition (ICP-OES inductively coupled plasma optical emission spectrophotometry)

The chemical composition was determined by Inductively Coupled Plasma Optical Emission Spectrophotometry (ICP-OES). 0.05 to 0.1mg of instrument segments were mixed with 1.75mL of hydrochloric acid (HCl) at 36.5-38% (Macron, Fine Chemicals®, Radnor, PA, USA), 5.75mL of nitric acid (HNO_3) at 64.5-66.5% (JT Baker) and 2mL of 48-51% hydrofluoric acid (HF) (Analytyka®) in EasyPrep Plus CEM ™ containers. The digestion was enhanced by microwave (Mars 6 CEM™) with a ramp of 15min and power of 900 to 1050kW. The digestion time was 30min at 210ºC. After that, the solutions were neutralized with 5mL of boric acid (H_3BO_3) at 4%v/v and gauged with 50mL of 2%v/v nitric acid. The final solution was analyzed to determine the Ni, Ti, and Au concentrations using the ICP-OES Instrument at wavelengths of 231,604, 336,121, and 242,795nm (iCAP 7400 Duo, Thermo Scientific™, Waltham, MA USA).

Statistical analysis

Statistical analysis of the six measurements per sample was performed using the SigmaPlot v13.1 software, where the Normality Test was performed, where the normality of the data was determined. Subsequenly, the Student "T" test was performed to determine a significant difference between the means of the two groups for each evaluated system considering a value of $p<0.05$.4

Results

Thermal Characterization (Differential Scanning Calorimetry-DSC)

The thermograms obtained from DSC of the NiTi instruments during the first heating and cooling cycle are observed in Figure 1. In them, the evolution of the transitions of the phases that the instruments undergo is observed, as well as the enthalpy of the transformation that is determined by the integration of the area under each one of the peaks presents, where the two phases coexist (17). In the heating cycle of rotating systems (Figure 1 A and B), both systems exhibited an endothermic peak associated with the transition of the austenite phase (As). For the Vortex Blue instrument, the transition begins at 22.94ºC and for the V-Taper 2H instruments at 13.77°C, finishing at 41.52 and 49.70°C respectively (Af). Likewise, we can observe that the enthalpy of the transform varies according to the type of instrument. About the cooling process, we observed the transformation from the austenite phase to the martensite phase, for the V-Taper 2H system it presents two exothermic peaks. Giving clues that the phase transition from austenite to martensite occurs in two steps. While the Vortex Blue system shows a transition in a single step, starting the martensite phase (Ms) at 43.02ºC and ending (Mf) at 17.69ºC.

In the case of the reciprocating systems, in Figure 1 C it was observed that both systems exhibit an endothermic peak during the heating cycle at 31.58°C (As) for the WaveOne Gold system, and 29.39°C for the Reciproc Blue system, which ends at 58.06°C and 53.81°C respectively. During the cooling process, an exothermic peak associated with the so-called inverse transformation from austenite to martensite, where the WaveOne Gold system presents a martensite phase start temperature (Ms) of 57.19ºC while Reciproc Blue shows started at 55.65ºC. This transition of the martensite state (Mf) ends at 31.54ºC for Wave One Gold and 19.11ºC for Reciproc Blue. Concerning the enthalpy of transformation, it was determined to be 3.63J/g and 4.56J/g respectively. As well as a slight shoulder within the peak of the Reciproc Blue transition is observed, indicating that transformation gives two different populations.

Structural characterization (X-ray diffraction-XRD)

Figure 2 shows X-ray diffraction patterns of the instruments evaluated, where the transition process of the phases and the complexity of the region of 40 to 45° in 2θ can be observed. Two intense peaks are easily seen in this region, one around the 42.4 in 2θ which is associated with the (110) plane, as well as the characteristic peak of the intermediate premartensitic phase (R phase) that can occur during the transition of the Austenite phase (B2) and Martensite (B19´). Such peaks are observed at 42.7° in 2θ, with which is associated with the plane (300), and at 45 and 62° with lower intensity. Likewise, the diffraction patterns were analyzed using the ICDD database.

Morphological Characterization (Scanning Electron Microscopy-SEM)

The design parameters of the most relevant rotary and reciprocating instruments such as diameter, taper, helical angle, and pitch were determined by analyzing the micrographs using the ImageJ software. The results obtained are reported in Table 1. Some images obtained with the backscattered electrons detector at different magnifications are shown in Figure 3 for the different evaluated systems.

Chemical Composition (ICP-OES inductively coupled plasma optical emission spectrophotometry)

The analyzes obtained by ICP-OES provide information on the content present in the Ni/Ti alloy used in the manufacture of endodontic instruments. The obtained results for the rotary instruments indicate that there is no statistically significant difference between the evaluated rotary systems, in terms of Ti content (p=0.666), but there is a significant difference in terms of Ni content (p=<0.001). Furthermore, the atomic ratio of Ni:Ti was determined to be 1.16:1.00 for the Vortex Blue instruments and 1.03:0.98 for the V-Taper 2H instruments. Likewise, the Student's "T" test in the reciprocating systems indicate that there is no statistically significant difference between the rotary systems evaluated in terms of the Ti content (p=0.129), but there is a significant difference in terms of the content. Ni ($p \leq 0.001$). While the atomic ratio was 1.08:0.90 of Ni:Ti for the Reciproc Blue instruments and 0.97:0.89 of Ni:Ti for the WaveOne Gold instruments, 0.11±0.02%w/w of gold was detected.

Discussion

The root canal treatment has been revolutionized by advances to the rotary instruments with nickel-titanium alloy with shape memory, which present high mechanical resistance and ductility. However, the mechanical stresses to which endodontic instruments are subjected differ according to the working movement. Therefore, the flexibility and resistance of the instruments should vary according to the clinical needs. Thus, currently there is a variety of systems from different commercial brands that offer their characteristics depending on the chemical composition and heat treatment to which the instruments were subjected, which give them a certain crystallographic arrangement and conditions in which the phase transformations may occur. NiTi alloys with cuasi equivalent atomic ratio exhibit phase transitions from martensite to austenite and viceversa, either directly or in two steps, which gives the instrument greater flexibility and mechanical resistance, and thus benefiting the area of endodontics. These transitions can be induced by stress or temperature, and the beginning and end of the transition depend on the Ni concentration and the heat treatment applied to the instruments during their manufacturing process (18,19). In this context, differential calorimetry scanning allows determining the temperature ranges of the temperature-induced transitions, and the enthalpy associated with the transition, which corresponds to the area under the curve of each of the exothermic and endothermic peaks (18). About the enthalpies determined for each of the reciprocating instruments, they were lower than the enthalpies observed in the rotating systems. This could be explained based on the instrument's requirements during its operation. Since, during canal instrumentation with reciprocating systems, the instruments will have a greater ability to recover from the martensitic phase by releasing stress during the counterclockwise movement of the instrument, if the stress does not exceed the elastic limit of the material. This could explain why reciprocating movements extend the useful life of a NiTi instrument and its fatigue resistance compared to the continuous rotational movement (20,21). Because rotating systems are subjected to continuous stress and increasing enthalpy of transition could help increase the cyclical fatigue resistance of the material. As it occurs with the V-Taper 2H instrument, which presents a double exothermic transition during the cooling process, associated with an intermediate rhombohedral phase known as the R phase, which corresponds to a second-order transition and is observed before the austenitic transformation which is characterized by high cyclic stability and therefore increases the useful life of the instrument (4,8). This transformation of the R phase has been reported to be induced by various treatments, including thermal cycling, annealing after deformation, aging of Ni-rich alloys, the appearance of a dislocation network, Ni_4Ti_3 precipitates or the addition of ternary elements (1,8,9). On the other hand, Vortex Blue instruments show a transformation in a single step (austenite-martensite). In addition, it is also observed that the temperatures at which the thermal transition begins in the reciprocating systems (As) are higher than those at ambient temperature (20°C), so it is inferred that these rotating instruments are in a martensitic state at the beginning of endodontic treatment, that is, they are ductile and flexible. However, this does not exclude the possibility that the instruments can go to the martensitic state through a stressful process that occurs during the instrumentation process in a clockwise movement to favor the cutting process. While, in the case of rotary systems, the starting temperature of said phase transition (As) is slightly below room temperature or earlier as in the case of V-taper-2H. Therefore, these rotary endodontic instruments evaluated are in a phase transition zone, where the different crystalline structures (martensite-austenite) coexist.

About the results obtained by ICP, it was determined that they contain Ni and Ti. A statistically significant difference was not found between the rotary and reciprocating systems evaluated in terms of Ti content, but a significant difference in terms of Ni content, which has been reported to play a fundamental role in the structural arrangement of the crystalline phases and imparting different mechanical behavior. Regarding the NiTi phase diagram, it has been reported that alloys with pseudoelastic applications must use Ni-rich alloys with Ni content of around 50.7at% (3,4,7). It should be noted that small traces of Au were detected in WaveOne Gold systems, which could be associated to be the result of the patented Gold treatment consisting of a light coating of this material, (12,13) which was also appreciated in the specific EDS analyses on the surface of this material (Figure 4R).

The interatomic arrangement or the crystalline structure of the instruments confers various mechanical properties to the instruments, which can be evaluated by X-ray diffraction. From this perspective, it should be noted that according to the results previously obtained by DSC, the endodontic instruments that were evaluated at 30°C, they are in a transition zone. Therefore, it is evident that peaks associated with different crystalline planes can be seen. For example, the most predominant peak observed in the austenite phase corresponding to the (110) plane is observed at 42.2° to 2θ, and in the case of the V-Taper 2H rotary instruments, we can confirm that the instrument passes through martensite to austenite through an intermediate phase called R. In addition, the standard JCPDS card coincides with Ni_4Ti_3 (01-078-4619), which has been mentioned in the literature as precipitates that induce this crystalline phase, as well as with the NiTi standard (01-076-7517) that corresponds to an equiatomic alloy. For all this, we can define that the parameters in terms of design, heat treatments, chemical composition, and the structural arrangement of the atoms are responsible for the different behaviors of the instruments, which have been modified over time to offer more efficient and safe instruments during root canal treatment. About the cross-section of the instruments, a convex triangular shape is characteristic of the Vortex Blue instrument, which will give it greater resistance compared to the triple helix design of the V-Taper 2H instrument (22,23). While in the reciprocating instruments their cross-sectional design favors the Reciproc Blue instrument with the "S" shape over the rectangular parallelogram shape of the WaveOne Gold system because the S-shaped sections are stronger than rectangle-based instruments (22,24,25).

Conclusions

The endodontic instruments evaluated are made of a nickel-rich alloy, which gives them pseudoelastic properties. Heat treatment modifies the phase transitions and, consequently, the flexibility and ductility properties. The type of heat treatment applied depends on the intended movement.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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TABLES AND FIGURE LEGENDS

TABLE

Table 1 Characteristic values of the design parameters of the instruments.

Parameter	Rotating Systems		Reciprocating Systems	
	Vortex Blue ®	V-Taper 2H ®	WaveOne Gold ®	Reciproc Blue
Helical angle [°]	17.87	21.23	21.68	29.58
Pitch [mm]	0.54	0.89	0.80	1.17
Tip and transition angles [°]	29.30	37.77	15.29	33.50
Diameter and taper	25/0.04	25/0.08	25/0.07	25/0.08
Cutting Angle	Negative	Positive	Positive	Positive

FIGURE

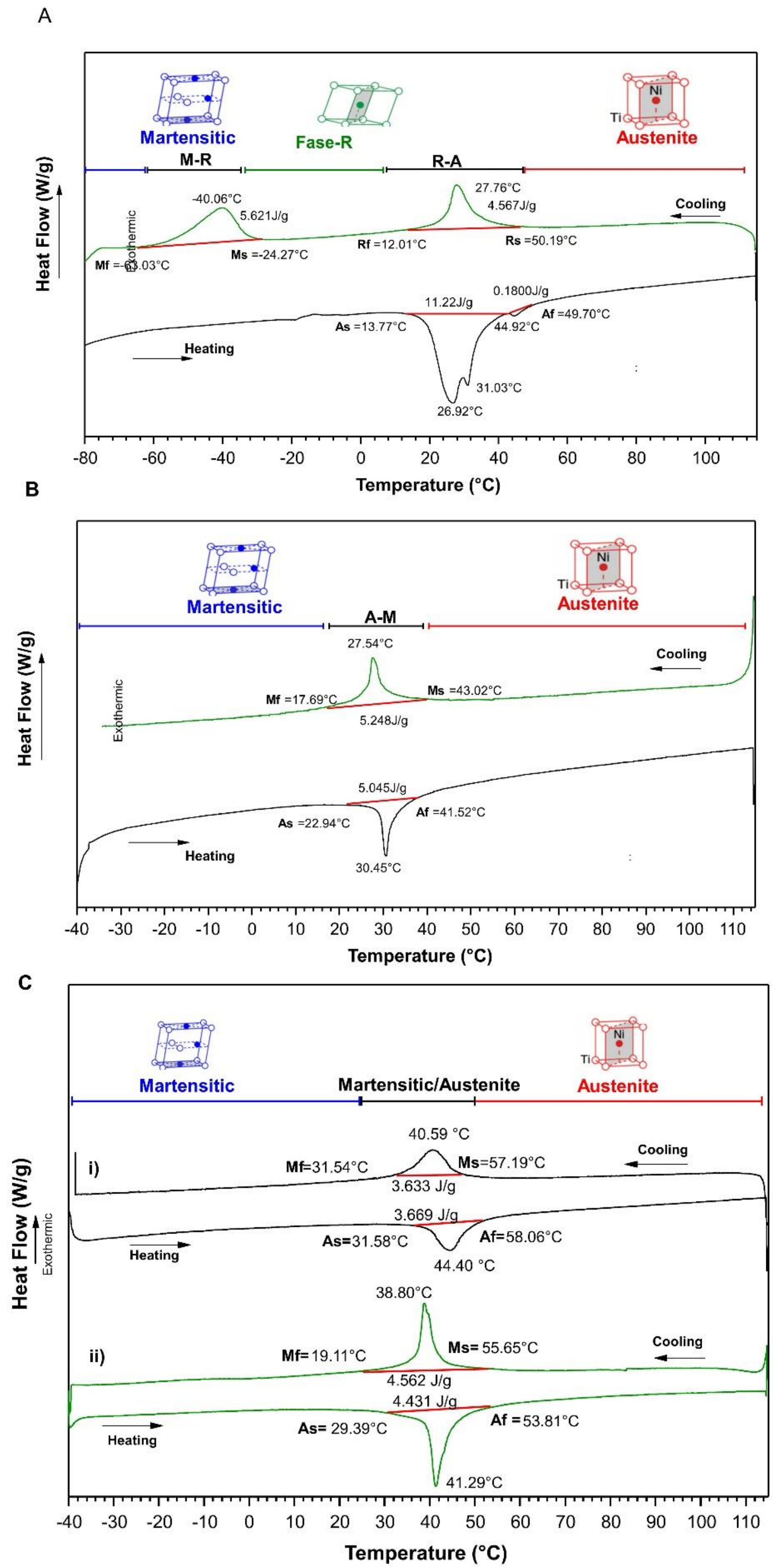

Figure 1: Thermogram. Rotating systems: a) V-Taper 2H and b) Vortex Blue; c) Reciprocating systems: i) WaveOne Gold and ii) Reciproc Blue.

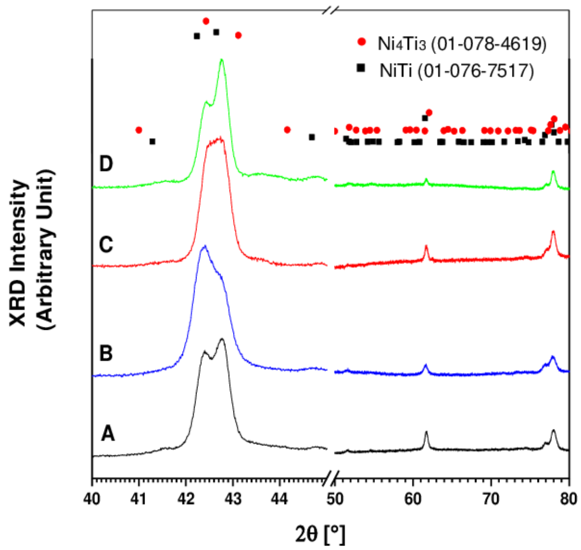

Figure 2: XRD spectra of a) Reciproc Blue, b) WaveOne Gold, c) Vortex Blue and d) V-Taper 2H.

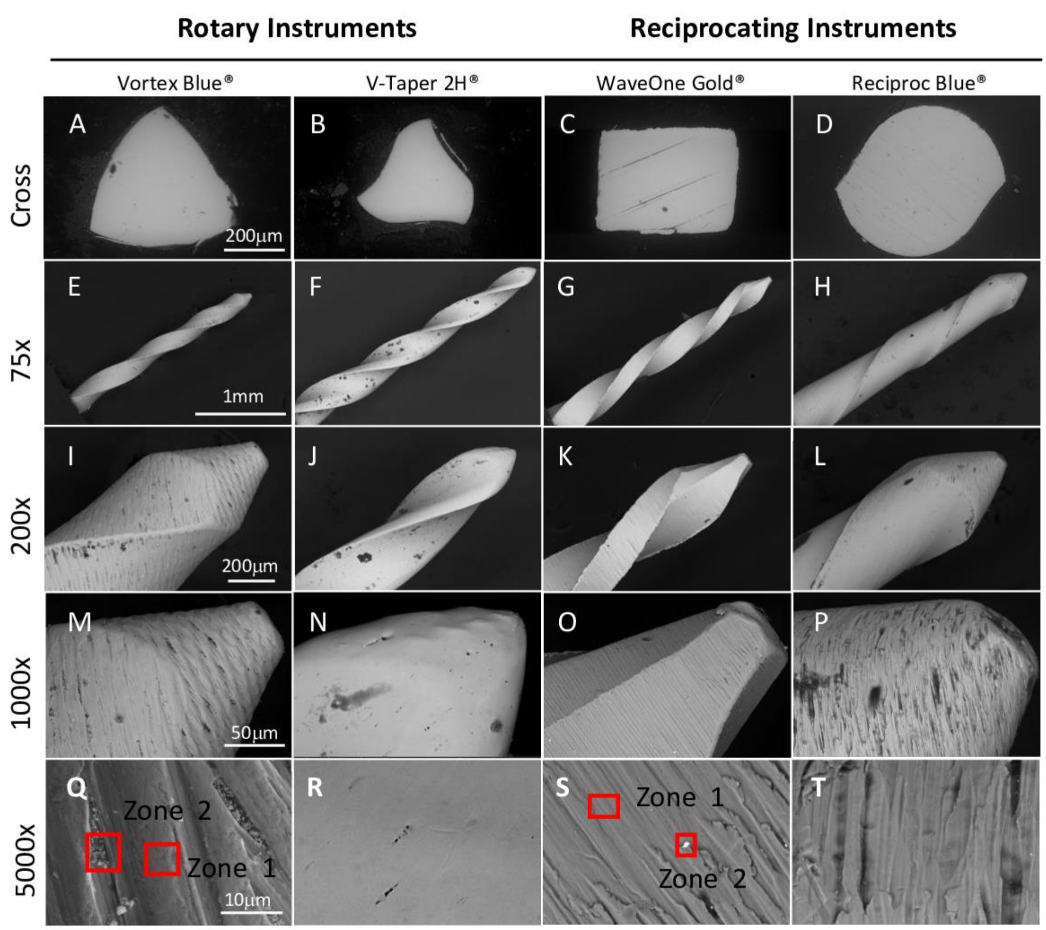

Figure 3: Backscattered electron microscopies. A-D) Cross section at 250x; E-H) Helical angle and step to 75x; I-L) transition angle at 200x; M-P) surface analysis at 500x where the homogeneity and morphology of the instruments are appreciated, with slight contrasts on some points of the surface of the material, giving indications of elements with different atomic numbers and Q-T) surface analysis at 1000x.

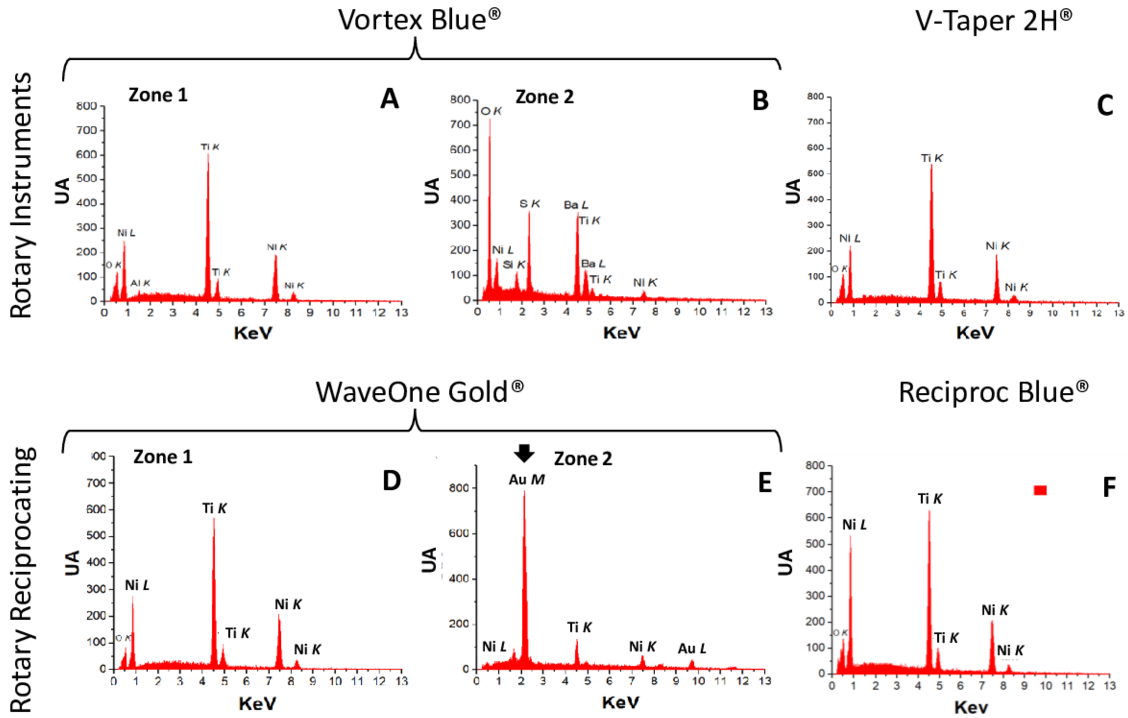

Figure 4: Energy-dispersive X-ray spectroscopy (EDS) analysis at different points of the instruments at 1000x. where you can see the main elements that make up the NiTi alloy. As well as some traces of heavier elements, possibly from the manufacturing process and other than the NiTi alloy.