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Comparison of Mechanical and Structural Properties of Nickel-titanium Alloy with Titanium-molybdenum Alloy and Titanium-niobium Alloy as Potential Metals for Endodontic Files

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ABSTRACT

Introduction: The objective of this study was to compare the mechanical and structural properties of the nickel-titanium (Ni-Ti) alloy already used in endodontics with titanium-molybdenum (Ti-Mo) and titanium-niobium (Ti-Nb) alloys to determine if these can be suggested in the manufacture of endodontic files. **Methods and Materials:** Orthodontic wires made of the different alloys were used. The previously mentioned alloys were characterized by energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD) and torsion tests. Cyclic fatigue tests were performed on a simulated canal with a curvature of 86° to 375 rpm. The fractured surfaces of the wires were observed by means of scanning electron microscopy (SEM). A Kruskal-Wallis test and U Mann Whitney test were used to determine significant differences in cyclic fatigue between groups. **Results:** In the mechanical tests, similar values of torsion were found for the three alloys. In XRD, the Ti-Nb showed less structural changes. In the cyclic fatigue test, Ti-Nb was found to be significantly more resistant with respect to Ni-Ti and Ti-Mo. **Conclusion:** Based on our *in vitro* study, Ti-Nb is suggested as a possible alloy for the manufacture of rotary files due to its impressive properties.

Keywords: Cyclic Fatigue; Nickel-titanium; Rotary Files; Titanium-molybdenum; Titanium-niobium

Introduction

One of the purposes of the root canal treatment (RCT) is to eliminate pulp remains and to shape the root canal through biomechanical cleaning and shaping. Currently, most endodontic files used to accomplish such goals are made of nickel-titanium (Ni-Ti) alloy. In this sense, a great variety of studies have been performed to characterize Ni-Ti files, both in metallurgical characterization and in mechanical tests [1, 2]. Despite the plethora of analytical studies performed on Ni-Ti files, sudden fracture may still occur clinically, given the fact that in many cases it is difficult to detect signs of alloy deformation [3, 4]. Therefore, it is important not only to perform thermal treatment to the Ni-Ti alloy in order to improve its mechanical properties [5], but also to investigate different alloys that may have useful properties for the manufacture of endodontic instruments.

In orthodontics, for example, the use of arch wires made out of different alloys is significant, as exemplified by the titanium-molybdenum (Ti-Mo) alloy, commonly known as β-Titanium or TMA®. This alloy was introduced to orthodontics in 1980 by Burstone *et al.* [6], and it is employed at any time during the orthodontic treatment [6]. As a matter of fact, it has been widely used because it exhibits an intermediate modulus of elasticity between Ni-Ti and stainless steel. Likewise, the titanium-niobium (Ti-Nb) alloy is also commonly used in different biomedical areas with different percentages as well as alloys with different chemical elements [7-9]. In orthodontics, Ti-Nb based alloy has been reported like an alloy useful in a smooth, continuous tooth movement [10] In addition, it could be used in nickel allergy patients [11] One commonly used Ti-Nb based alloy is Gunmetal®, which is a wire used during orthodontic treatment due to showing a favorable tension distribution pattern [12].

Scanning electron microscopy (SEM) is used in endodontics to characterize materials, and this technique can be coupled with a probe for chemical analysis, as energy-dispersive X-ray spectroscopy (EDX), which provides additional information about the chemical composition of the material. In addition to SEM, X-ray diffraction (XRD) has been used as a non-destructive physicochemical technique to determine atomic positions in the study materials, crystalline phases, and network parameters of the structure [1].

The mechanical properties of endodontic files are also studied employing torsion and cyclic fatigue tests [13]. Of note, in endodontics the torsional fracture of an instrument occurs when the file is in continuous rotation and comes into contact with the walls of the root canal. At that moment, part of the instrument is locked and the motor that drives it keeps on operating; the instrument cannot be released and it exceeds its torsion limit leading to a fracture [14].

In endodontics the fracture of flexural instruments is generated when the file rotates freely within a curvature, constantly generating cycles of tension (external area of the curve) and compression (internal area of the curve), thus weakening the material and generating the propagation of microcracks [15]. The latter process leads to the point of maximum flexion and then fracture of the instrument [2].

Likewise, the endodontic instruments must be resistant to cyclic fatigue by having sufficient flexibility to rotate in curved root canals. Of note, to date, there is no international specification or standard for testing cyclic fatigue resistance of endodontic instruments, so there are multiple reports with varying methodologies [2].

The objective of the present study is to evaluate the mechanical and structural properties of the conventional Ni-Ti alloy wire used in endodontics and to compare it with Ti-Mo and Ti-Nb wire alloys using XRD, and cyclic fatigue testing. The null hypotheses were that there are no differences both in cyclic fatigue and in the mechanical properties evaluated. The impact of this work is that it will determine if these alloys can be potentially used in the manufacture of endodontic instruments for biomechanical instrumentation in RCTs.

Materials and Methods

Selection of material

Orthodontic wires with rectangular conformation in their transversal design were used in three different types of alloys: Ni-Ti of 0.40 mm (0.016 inches) × 0.55 mm (0.022 inches), Ti-Mo 0.40 mm (0.016 inches) × 0.55 mm (0.022 inches) and Ti-Nb 0.45 mm (0.018 inches) × 0.55 mm (0.022 inches).

Samples were randomly obtained from the wires corresponding to each alloy, and element analysis was carried out using EDX BRUKER-QUANTAX (Bruker Nano GmbH, Berlin, Germany) in a SEM TESCAN VEGA 3 (Libušina tř, Brno-Kohoutovice, Czech Republic).

Torsion test

With 150 mm long wires, three replicates were made for each of the three alloys, making a direct measurement on the torque transducer, with a reference standard of 500 Ncm.

XRD

XRD was conducted in order to detect the crystalline phases present in six samples: three unloaded samples (one from each alloy) and three samples subjected to torsion (one from each alloy). Measurements were performed using a Panalytical Empyrean diffractometer (Malvern Panalytical, Malvern, UK) with a copper anode (Cu K λ = 1.5406 nm), step size of 0.02 s and 2θ range from 30° to 80°. The choice of this 2θ measurement interval was due to the fact that, in the case of the prepared samples, it is in this interval that the PDF-Card 44-1294 used presented some coincidences, since not all the reflections of the associated diffraction peaks appear in a greater value of 2θ. Thus, depending on the number of reflections, the interval 2θ can be used, so its choice depends on the sample, as has been reported previously with a 2θ interval from 20° to 90° [16, 17].

Cyclic fatigue test

The assembly of a numerical control device was performed to decrease and calibrate the speed of the continuous rotation of each sample, which was at 375 revolutions per min (rpm). This assembly was made directly on a bore that had a speed control system. An optocoupler with an Arduino® circuit was used to measure the revolutions. This device was programmed with an rpm frequency meter on the card of the Arduino microcontroller board (Arduino MEGA 2560, Ivrea, Italy) by displaying directly on the Arduino software (Arduino IDE 1.0.x, Ivrea, Italy), which recorded the rpm reading of the serial monitor. To perform the cyclic fatigue test, the wire was confined to a simulated stainless steel canal (length: 30 mm, diameter: 1.5 mm, curvature: 86°) [15]. Each wire had a length of 50 mm and 10 repetitions were made per group [13]: Group 1: Ni-Ti alloy; Group 2: Ti-Mo alloy, and Group 3: Ti-Nb alloy.

Finally, three samples fractured in the cyclic fatigue procedure were randomly obtained from the wires used and then analyzed under SEM. This was done in order to establish the main morphological characteristics of the fractured surfaces.

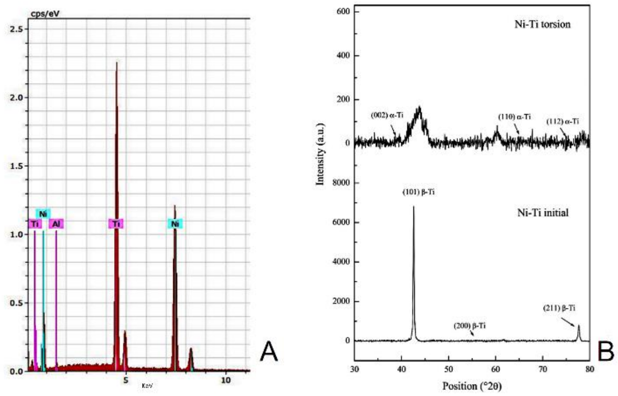

Figure 1. *A)* Energy Dispersive X-ray spectroscopy (EDX) spectrum for Ni-Ti alloy; *B)* X-Ray Diffraction (XRD) diffractogram for Ni-Ti alloy

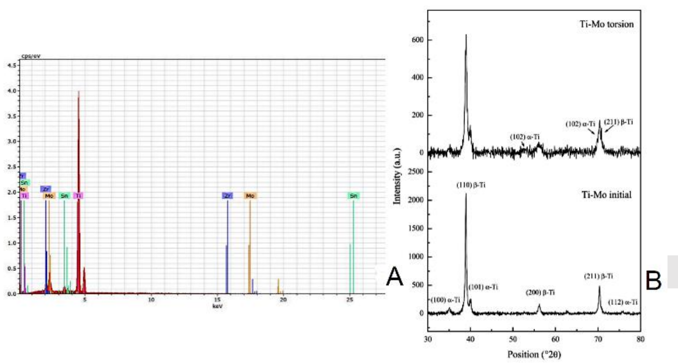

Figure 2. *A)* Energy Dispersive X-ray spectroscopy (EDX) spectrum for Ti-Mo alloy; *B)* X-Ray Diffraction (XRD) diffract gram for Ti-Mo alloy

Statistical analysis

The data were initially analyzed using the Shapiro-Wilk test in order to confirm a normal distribution. Then, a Kruskal-Wallis test was performed, and a Mann-Whitney U test was used to determine which group was different. The level of statistical significance was 5%, and the software used was Stata version 12 (StataCorp LP, College Station, Texas, USA).

Results

Element composition in atomic percentages of each wire can be seen in Table 1 and Figures 1A, 2A, and 3A. Overall, Ni-Ti is an equiatomic alloy, and Ti-Mo, and Ti-Nb alloys present a greater percentage of Ti than Mo and Nb, respectively. In EDX, the elements in atomic percentages that were observed are shown in Table 1 and Figures 1A, 2A and 3A.

Torsion test

Results of the torsion test are presented in Table 2. Similar torque values between the three alloys can be seen, with highest value for Ni Ti alloy, followed by Ti-Nb alloy.

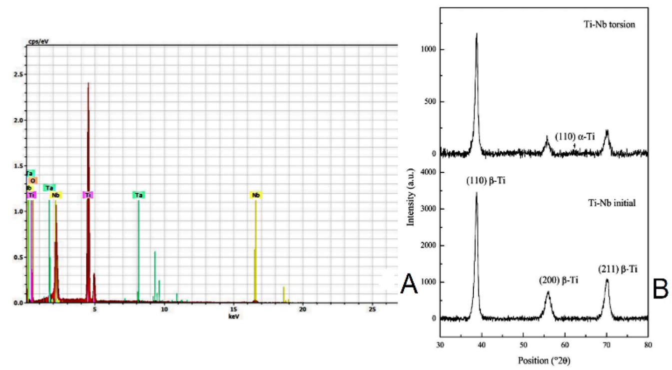

Figure 3. *A)* Energy Dispersive X-ray spectroscopy (EDX) spectrum for Ti-Nb alloy; *B)* X-Ray Diffraction (XRD) diffractogram for Ti-Nb alloy

XRD

In the diffractogram made to the Ni-Ti alloy (Figure 1B), it exhibited the plane (101), which was the most intense, as well as the planes (200) and (211). These planes were associated with the β-Ti phase (PDF-Card 44-1294) [18]. When comparing with the diffractogram of torsion test, the peaks of the β phase decreased in intensity, but the α-Ti phase was formed (PDF-Card 44-1288) [18], with planes (002), (110) and (112). With regard to the Ti-Mo alloy, in the diffractogram shown (Figure 2B) the other Ti diffraction peaks, the plane (100) and the (112), were associated with the phases α and β of the Ti, and during the torsion, more diffraction peaks of the α-Ti phase appeared. In the diffractogram corresponding to the Ti-Nb sample (Figure 3B), the diffraction peaks corresponding to the β-Ti were observed with the planes (110), (200) and (211), while after the torsion, the plane (110) of the α-Ti appeared.

Cyclic fatigue test

Ten repetitions per alloy were performed. Ti-Nb based alloy presented significantly more resistance to fatigue fracture than Ni-ti alloy and Ti-Mo alloy *(P*=0.000).

The Kruskal-Wallis and Mann-Whitney U tests were performed to determine the difference in the number of cycles between groups (*P*=0.002), which allowed the conclusion that at least one of the groups was different and that the alloy that presented a significantly higher number of cycles of fracture was Ti-Nb, followed by Ni-Ti and with a significantly lower value for Ti-Mo (*P*=0.001) (Table 3). With regard to the analysis of the surface exhibiting fracture defects, Ni-Ti wire alloy showed a smooth zone at the starting point of the defect with the presence of microcracks and a fibrous zone with abundant dimples, less plastic deformity and fewer shear lips (Figure 4A, 4B). The Ti-Mo alloy showed more shear lips when compared to Ni-Ti alloy, and it exhibited a smooth zone with microcracks, less dimples and more plastic deformity likely associated with a ductile fracture (Figure 4C, 4D).

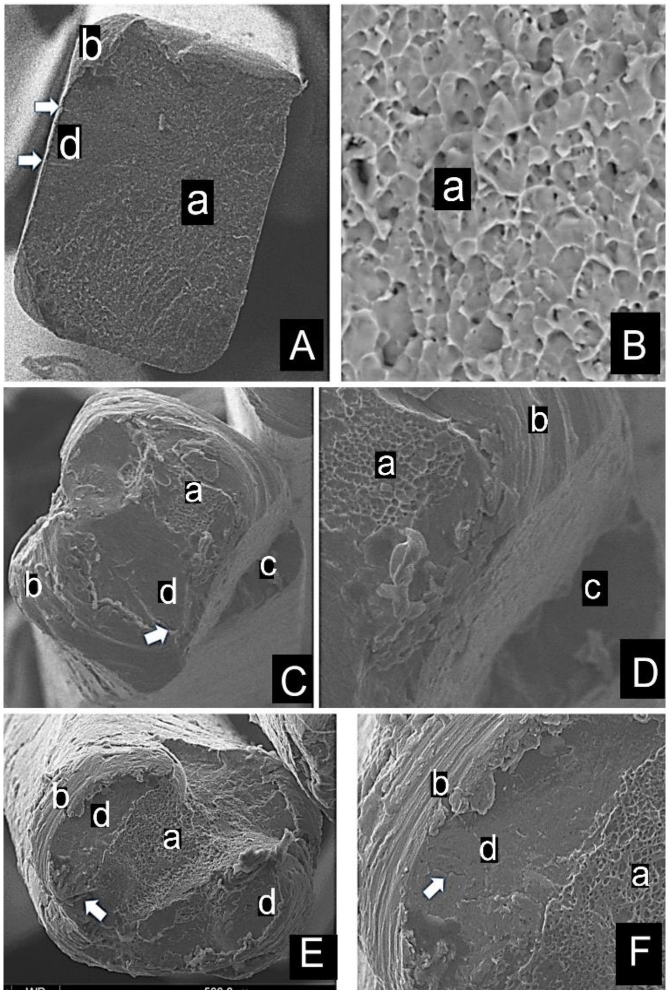

Figure 4. SEM micrograph of the fractured cross-sectional surfaces of wires of Ni-Ti, Ti-Mo and Ti-Nb alloys. *A*) Typical image of the fractured surface of a conventional Ni-Ti alloy (original magnification 500×) shows a fibrous zone with dimples characteristic of overload (a), few shear lips (b), and zone of smooth fatigue (d) with beginning of microcracks on the edge (white arrows); *B*) Central zone of the fibrous region of (a) (original magnification 2600×) shows flattened dimples characteristic of a fracture with little plastic deformation and limited ductility (a); *C*) Image of the fractured surface of a Ti-Mo alloy wire (original magnification 500×) shows greater amount of shear lips observed than in (a) wide smooth zone typical of fatigue (d) with the presence of microcracks (white arrows), coarse slip band (c) and scarce fibrous zone with few dimples (a); *D*) Increase of a region of (c) (original magnification 200×) shows the dimples of the fibrous zone (a), the coarse slip band (c) and the shear lips (b) observed in detail; *E*) Image of the fractured surface of a Ti-Nb based wire (original magnification 500×) shows evident plastic deformation, fibrous zone with dimples characteristic of overload and ductile fracture (a), shear lips (b), and zones of smooth fatigue (d) with presence of microcracks (white arrows); *F*) Increase of a region of (e) (original magnification 100×) shows elongated dimples characteristic of torsional overload (a), shear lips (b) and microcracks (white arrows) in the smooth area of fatigue (d)

Lastly, Ti-Nb alloy showed more shear lips than both Ni-Ti and Ti-Mo alloys, and it presented abundant smooth zones with microcracks and several dimples in the central zone with great plastic deformity reflecting a fatigue fracture predominantly ductile with torsional overload (Figure 4E, 4F).

Discussion

According to the present results, the Ti-Nb-based alloy is more resistant to cyclic fatigue than the Ni-Ti and Ti-Mo alloy. This cyclic fatigue resistance result may be correlated with the relatively low modulus of elasticity of Ti–Nb [19, 20], which could eventually be an advantage for the preparation of curved root canals. With the results of this study, we may suggest the use of Ti-Nb as an alloy for the manufacture of rotary files to be used in RCT. Of note, this alloy has been widely used in other biomedical areas with some modifications: mechanical and chemical [7].

The number of cycles at fracture reported in the literature for different endodontic instruments may be higher than those recorded in the alloys in this study [21-23], but as in torsion, in the cyclic fatigue the design influences the resistance of the instruments [22, 24]

Murakami *et al.* [25] evaluated the resistance to fatigue of high cycles of Ti-Mo and Ti-NB alloy wires, and they found that there were no significant differences in fatigue resistance between the analyzed alloys [25]. This result does not agree with our results where the Ti-Nb alloy was significantly more resistant to fatigue than the Ti-Mo alloy. The discrepancy of results may be due to several factors. They evaluated fatigue at high cycles of the wires with the methodology used for engineering materials based on the use of a universal testing machine [25] ,while in the methodology of the present study, we evaluateD cyclical fatigue by simulating the behavior of the wires in an artificial root canal like in an endodontic treatment, which has not been previously reported in the literature for use in orthodontic wires.

Since the last decades of the twentieth century, it has been documented that both super elasticity and shape memory effect are among the physical properties that have been reported as advantageous for Ni-Ti alloys [26].

Recent research, however, has been conducted to improve the mechanical properties of Ni-Ti alloy by means of various thermal treatments [26-28]. Included among modifications obtained are decreasing its modulus of elasticity, which makes the alloy more ductile and with control of the shape memory effect [23].

The files manufactured with said alloys have been reported to have greater resistance to cyclic fatigue fracture than those manufactured using either conventional Ni-Ti alloy or M-Wire [23, 29].

Although Ni-Ti alloy files are still the subject of research and subject to improvement, it is still necessary to keep looking for new alloys that have superior properties than those under current use. For example, in this case we evaluate the Gummetal® alloy (Rocky Mountain Morita Corporation, Tokyo, Japan), mainly composed of Ti-Nb which presents mechanical properties similar to those of thermally treated Ni-Ti alloy, such as a minor modulus of elasticity and good ductility. These properties have been reported to give excellent resistance to cyclic fatigue [30] and could explain the results of this study.

Viana *et al.* [31] reported that the element composition of Ni-Ti alloy of endodontic files has 50.5% Ni and 49.5% Ti, similar to our results where we found 49.07% Ni and 49.80% Ti [31].

Goldberg *et al.* [32] reported the element composition of Ti-Mo based alloy: 77.8% Ti, 11.3% Mo, 6.6% Zr and 4.3% Sn [32]. Our study found the same elements, but a different amount of each element was observed: 89.41% Ti, 5.35% Mo, 3.72% Zr and 1.52% Sn. The difference may be caused by the method used in the analysis.

Nagasako *et al.* [19] reported the same elements observed in this study, like 73% Ti and 22% Nb [19]. In our study, we found 51.56% Ti and 15.80% Nb. The difference is possibly caused by other elements present in both cases such as O and Ta in different ratio.

Odegaard *et al.* [33] reported that the fracture torque for the TMA orthodontic wire was 1.53 N cm, and for the Ni-Ti orthodontic wire it was 2.21 Ncm [33], both values being below what we found in this study. Again, the discrepancy may be caused by the methodology. Our evaluation was carried out with a transducer torque simulating the entrapment of a section of the material within a root canal while Odegaard *et al.* [33] calculated the moments by adding fixed weights to a basket.

McGuigan *et al.* [4] reported that initial preparation with endodontic instruments with small tip diameter and small taper, such as Pathfile (Maillefer-Dentsply, Ballaigues, CH, Switzerland) or ProGlider (Dentsply Maillefer, Ballaigues, Switzerland), require a lower torque value (close to 2 Ncm) [4], which agrees with the values of resistance to torsion fracture found in this study. It can be suggested that Ti-Mo and Ti-Nb alloys could be useful in the manufacture of the above-mentioned type of files in endodontics.

In the diffractogram corresponding to the initial Ni-Ti alloy, the diffraction peaks associated to the β-Ti phase are well defined, and only such phase is shown. When performing the torsion test, a mixture of phases is generated, as also diffraction peaks belonging to the α-Ti phase appear. By decreasing the intensity of the diffraction peaks of the β-Ti phase, the crystallinity, product of torsion, decreases. In the diffractogram of the Ti-Mo alloy, the low amount of molybdenum makes it possible to observe more diffraction planes of titanium in the alloy. Comparing with the sample that was subjected to torsion, it was also observed that the initial diffraction peaks were maintained, but two additional diffraction peaks belonging to the α-Ti phase appeared, indicating that there is a structural change. The intensity of the diffraction peaks decreased with torsion.

Table 1. Atomic percentages of the elements present in the three alloys.

Ni-Ti		Ti-Mo		Ti-Nb	
Element	**At. %**	**Element**	**At. %**	**Element**	**At. %**
Ni	49.07	Ti	89.41	Ti	51.69
Ti	49.80	Mo	5.35	O	31.98
Al	1.13	Zr	3.72	Nb	15.80
		Sn	1.52	Ta	0.53

Nickel Titanium (Ni-Ti), Titanium Molybdenum (Ti-Mo), Titanium Niobium (Ti-Nb)

Table 2. Maximum torque values for the three alloys

Alloy	Maximum Torque Value
Ni-Ti	3.5 N cm
Ti-Mo	3.0 N cm
Ti-Nb	2.7 N cm

Nickel Titanium (Ni-Ti), Titanium Molybdenum (Ti-Mo), Titanium Niobium (Ti-Nb)

Table 3. Number of seconds of each repetition with a speed of 375 rpm.

Repetitions	Group 1: Ni Ti		Group 2: Ti Mo		Group 3: Ti Nb.	
	Seconds	**Cycles to fracture**	**Seconds**	**Cycles to fracture**	**Seconds**	**Cycles to fracture**
1	9.75	61	4.3	27	14.99	94
2	9.67	60	4.23	26	1503	94
3	10.8	68	4.84	30	14.25	89
4	9.81	61	4.96	31	15.11	94
5	9.78	61	4.47	28	15.05	94
6	8.9	56	4.33	27	15.23	95
7	9.92	62	5.17	32	15.13	95
8	9.95	62	4.33	27	14.86	93
9	9.5	59	5.66	35	14.93	93
10	9.88	62	4.27	27	14.95	93
Mean (SD)	61.2 (3.01)		29.0 (2.90)		93.4 (1.71)	

Nickel Titanium (Ni-Ti), Titanium Molybdenum (Ti-Mo), Titanium Niobium (Ti-Nb)

The results obtained for the Ti-Nb alloy demonstrated that an additional phase associated with the torsion force of the α-Ti phase was generated when the diffractograms were compared. The planes (110), (200) and (211) are maintained during twisting, but the intensity of such peaks decreases. The crystallographic structure of the sample is not greatly affected by the torsional stress. The presence of niobium does not increase the formation of diffraction planes of the alloy. That is to say, a smaller change of crystallinity is generated. Iacono *et al.* [34] in a study of twotypes of files such as HyFlex EDM (Coltene; Cuyahoga Falls, OH, USA) and HyFlex CM (Coltene; Cuyahoga Falls, OH, USA) also found a combination of phases in the reading of the diffraction peaks [34].

The SEM analysis of the fractured cross-sectional surfaces showed that the Ni-Ti wire presented zones of fatigue with microcrack-starting points on the edge. A similar phenomenon has been reported in rotary files of conventional Ni-Ti alloy, where the cutting edges act as stress concentrators favoring the start of microcracks [35]. Similarly, an overload zone with characteristic ductile fracture dimples is also observed, which agrees with what was previously reported for laboratory studies [36]. In the Ti-Mo alloy, shear lips and several zones of fatigue were observed with the presence of microcracks that would be the possible causes of the lower resistance to cyclic fatigue. A coarse slip band was also observed that agrees with what was reported by Sugano *et al.* [37].

The Ti-Nb alloy presents several zones of fatigue with microcracks, but due to its greater toughness, it shows a microcrack dispersion, which leads to decrease the fracture probability due to a dissipation of the microcrack energy propagation. The increase of the Ti-Nb toughness is possible because of its good ductility. Similar findings for the controlled memory alloy were previously reported [38]. In addition, the good ductility decreases the stress levels, which would explain in part the increase in the resistance to cyclic fatigue in a severe curvature as it was in this case. Finally, as well as in the Ni-Ti alloy, the surface treatments improve the resistance to cyclic fatigue [39]. More research in this topic is needed, where the use of these treatments could be proposed in alloys based on Ti-Nb in order to determine which of them could increase its resistance to cyclic fatigue and therefore may be useful in the manufacture of rotary files to use in root canal treatment in endodontics

One of the limitations in our study research were the dimensions in which Gummetal® was obtained was not equivalent to the other alloys analyzed. However, it has been reported that resistance to fatigue of rotating endodontic files has a close inverse relationship with the transverse area of the file [40, 41]. In addition, cyclic fatigue is a multifactorial process in which the pressure exerted on the file, the temperature, the root canal anatomy and the taper should be considered for relating to the clinical situation. Furthermore, manufacturing techniques like heat and surface treatments [22, 23, 39] affect resistance to cyclic fatigue.

Moreover, limitations should also be taken into account, like that the resistance to fatigue decreases significantly when the angle of curvature is greater [41] as is the case of the present study (86°). That is to say, although it would have been ideal to compare wires with equal dimensions, in our study, the wire of greater caliber was precisely Gummetal®, which despite the factors mentioned above, showed the greatest resistance to cyclic fatigue.

Conclusions

Based on this *in vitro* study the Ti-Nb alloy could potentially be an alternative metal for manufacturing endodontic rotary files. In our study, Ti-Nb registered the least amount of structural changes, together with appropriate resistance to torque and high resistance to cyclic fatigue.

Conflict of Interest: 'None declared'.

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