A NiTi rotary instrument manufactured by twisting: morphology and mechanical properties

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ABSTRACT

Objectives: The surface morphology of TF® endodontic instruments was studied using stereomicroscopy and scanning electron microscopy (SEM). Mechanical tests were done for flexibility and microhardness. **Methods:** Four tapers of TF® files were used (0.04; 0.06; 0.08 and 0.10 mm/mm). The stereomicroscopy associated with the AxioVision® program was used to measure the tip angle, the helical angle, the taper and the tip diameter of the instruments. SEM was used to identify surface defects due to machining and finishing. The flexibility and the microhardness were measured with bending and microhardness Vickers tests, respectively. **Results and Conclusion:** The analysis showed that the manufacturer complied with the values recommended by the ANSI/ADA standard number 28. The SEM results showed many surface defects and a distortion of the instrument helix. It was observed that the instrument flexibility changes with its taper. The forces to induce the phase transformation by stress on instruments with taper 0.04; 0.06 and 0.08 mm/mm were 100 gf, 150 gf and 250 gf, respectively. The values of Vickers microhardness of the instruments are compatible with rotary instruments manufactured by the machining process.

Keywords: Endodontic instruments. NiTi alloy. R-phase. Materials characterization. Mechanical tests. NiTi manufacturing methods.

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Introduction

In 1988, Walia et al[1] used a new metal alloy to manufacture endodontic instruments, the NiTi alloy. The instruments produced with this alloy had a lower Young modulus than the instruments made with stainless steel, thus allowing the endodontic treatment of cases with large root curvatures. The use of instruments made with stainless steel could make the treatment more difficult.

The first endodontic instruments of NiTi where manufactured by a machining process using burs. With the development of new NiTi alloys, the study of the mechanisms involved in the phase transformation and better control of the microstructure, it was possible to develop a new manufacturing method based on twisting. The TF® instruments (Twisted Files, California - USA) are manufactured by twisting. This new generation of instruments has better clinical properties.

In the present work the surface morphology of endodontic instruments manufactured by twisting was investigated and microhardness and flexibility measurements were performed. These properties are important to understand the clinical behavior and to develop new instruments.

Materials and methods

Morphology

The TF® endodontic instruments (Twisted Files, California) used in this study has a length of 27 mm, a tip diameter (D_o) of 25 mm. Three different tapers were used (0.04, 0.06 and 0.08 mm/mm).

The tip angle, the tip length and the taper were determined with an optic microscope Zeiss with a pixeLINK camera model PL- a662 and a light source Zeiss 1500 LCD. The taper was determined with an amplification of 1.6X. The other dimensions were quantified with an amplification of 5X. All dimensions of the instruments were determined with the program AxioVison 4.4®. Five instruments with each taper were investigated.

Bending tests (at 45°)

The bending tests were performed with an apparatus connected to a universal material testing system EMIC DL1000 (EMIC Equipment, Brazil). A 20 N load cell was used to measure the force necessary to bend the tip of the instruments by 10°, 20°, 30° and 45°. The tests were performed according to ADA standard 28, with the force applied 3 mm from the tip of the instrument.

Vickers Microhardness

For microhardness testing, the instruments were embedded in epoxy resin. The fixation cable was parallel to the recipient base with the purpose of keeping the central longitudinal surface outside of the resin after polishing. The instruments were prepared with sandpaper 200, 300, 400, 600 and 1200 and polished with alumina particles of 0.5 μm.

The Vickers indentations were made with 100 gf during 15 s using a microdurometer Bhueler model 1600-5300. Five indentations were made in the working part and five in the neck of each specimen.

Scanning electron microscopy (SEM)

Two instruments of each taper were submitted to SEM (JEOL, LSM 5800LV) to evaluate the morphologies of the cutting edge, the tip and interface of the neck region with the fixation cable.

Statistical analysis

The data of the bending tests and the Vickers microhardness were analyzed statistically by the Kruskal-Wallis method and complemented with the Student-Newman-Keuls multiple comparison test to compare the tapers. The microhardness at the neck region was compared applying the Mann-Whitney test. The level of significance of all analyses was 5%.

Results

The results of the statistical analysis are shown in Tables 1 and 2. The bending testing results are shown in Table 3. Figure 1 shows a mean curve obtained from 10 bending tests performed in instruments with taper 0.06. The tests for other tapers showed similar curves.

The curves show a slope change that is attributed to a phase transformation. The values of the forces necessary to bend the instruments by 10°, 20°, 30° and 45° are shown in Table 2 and the forces necessary to induce phase transformation by stress are shown in Table 3.

Statistical analysis (Kruskal-Wallis test) demonstrated that there was a significant difference between instruments with different tapers (P < 0.00001). Then, the Student-Newman-Keuls multiple comparison test revealed that the instrument of taper 0.04mm/mm is more flexible than instruments of tapers 0.06 and 0.08 mm/mm. Moreover, the instrument of taper 0.06 mm/mm proved to be more flexible than the instrument of taper 0.08 mm/mm.

The Vickers microhardness average values at the neck region and at the working region of the instruments are shown in Table 4.

The Vickers microhardness results for each taper were submitted to the Mann-Whitney test and there was no significant difference between the values in the neck region and in the working region for all instruments ($p > 0.05$).

Table 1. Tip angle, Tip length (L) and taper of the instruments.

Instrument	Taper 0.04	Taper 0.06	Taper 0.08	Taper 0.10
Tip angle	26.56 ± 4.39	32.41 ± 7.59	32.39 ± 13.89	25.48 ± 4.92
L (mm)	0.24 ± 0.011	0.25 ± 0.013	0.24 ± 0.007	0.26 ± 0.012
Taper	0.039 ± 0.0029	0.061± 0.0016	0.077 ± 0.001	0.099 ± 0.0022

**Table 2.** Average values of the maximum forces to bend at 45º (gf) and respective standard deviations.

Instrument	Taper 0.04	Taper 0.06	Taper 0.08
10º	67.82 ± 7.02	130.7 ± 17.21	179.7 ± 20.62
20º	92.26 ± 4.36	183.9 ± 16.17	295.2 ± 26.27
30º	120.3 ± 7.27	247.5 ± 20.61	390.3 ± 23.15
45º	131.7 ± 9.43	263.6 ± 23.18	400.7 ± 23.88

Table 3. Average forces for phase transformation by stress.

Instrument	TF 0.04 mm/mm	TF 0.06 mm/mm	TF 0.08 mm/mm
Average force	100 gf	150 gf	250 gf

Table 4. Vickers microhardness of the instruments.

| Instrument	HV neck region	HV working region
0.06	272.4 ± 31.6	291.2 ± 24
0.08	292.8 ± 33.8	293 ± 17
0.10	315.5 ± 33.7	279 ± 10.7
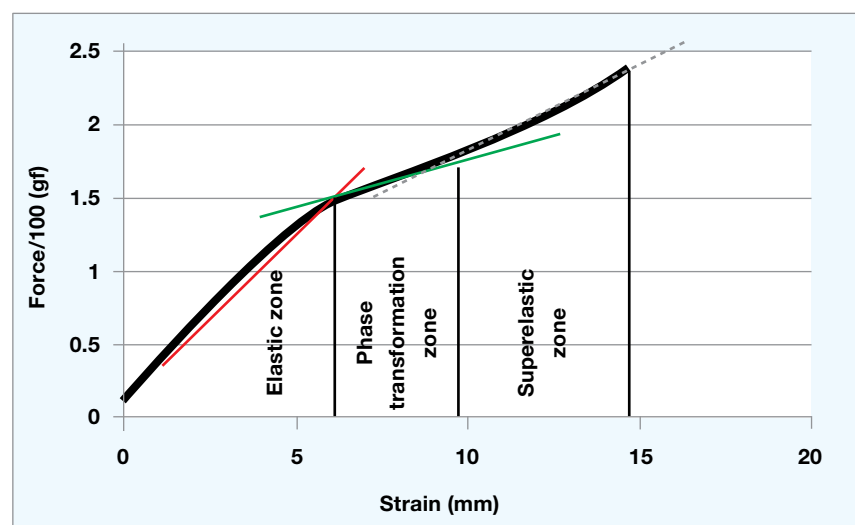
Figure 1. Mean curve for taper 0.06 mm/mm TF® files. The red line represents the elastic region, the green line phase represents the transformation region and the dashed line the superelastic region.

The microhardness values were also analyzed by the Kruskal-Wallis test. The statistical analysis confirmed that there was no significant difference among the groups (p=0.658). It is possible to conclude that the Vickers microhardness is independent of the taper and instrument region tested.

Surface analysis showed manufacturing defects in all instruments analyzed (Fig 2).

Figure 2 shows grooves produced in manufacturing process. It is possible to see the drawing tool marks along the longitudinal direction. All the samples had microcavities.
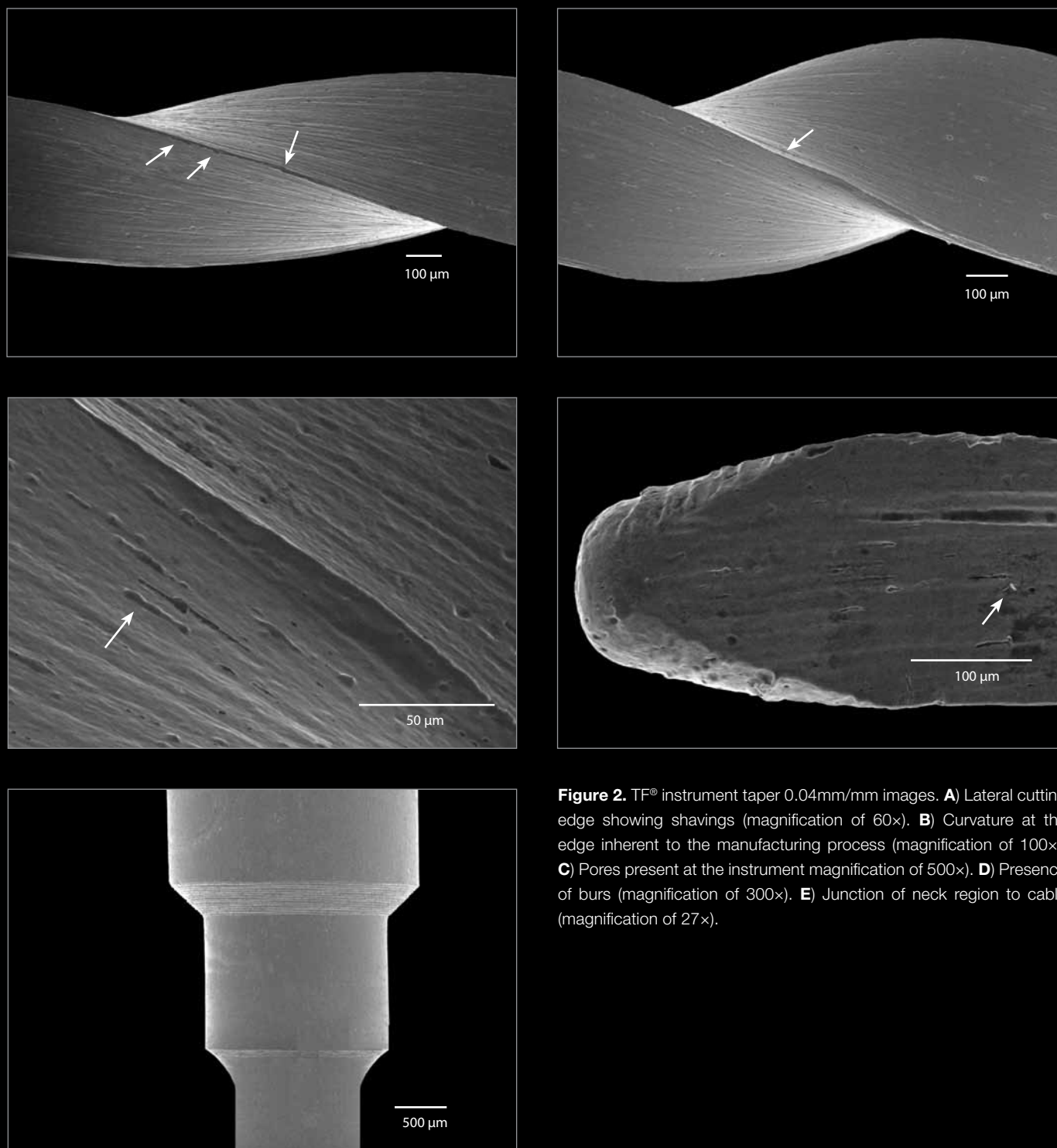
Figure 2. TF® instrument taper 0.04mm/mm images. **A**) Lateral cutting edge showing shavings (magnification of 60×). **B**) Curvature at the edge inherent to the manufacturing process (magnification of 100×). **C**) Pores present at the instrument magnification of 500×). **D**) Presence of burs (magnification of 300×). **E**) Junction of neck region to cable (magnification of 27×).

Discussion

Is important that the instrument tip has a good finishing and a transition angle that permits the introduction in the root canal. Small angles (less than 33º) can generate steps or deviations. The TF® instruments have a progressive tip angle that allows introduction in regions with a substantial curvature without deforming the canal, thus promoting a safe enlargement. The round tip can be classified as smooth.[2] Due this configuration, it is likely that the instrument will not cause damage to the root.

The instrument dimension D_o is determined by the diameter at the base of the tip basis, which serves as a reference during introduction. The value of D_o and the taper permits to determine the work diameter in a given canal region. A simple calculation can be used to determine what instruments can be used in sequence to perform an effective work. The diameter values of the tip bases (D_o) found on this study meet the ANSI/ADA standard number 28 recommendation. In the present work, we observed that the tip angles conform to the standard recommendations.

According to Thompson[3] the phase transformation of NiTi alloys does not show macroscopic changes when the application of an external force changes the microstructure. The bending tests showed a change in slope after an initial linear increase that resembled the Hooke law. This change is attributed to an austenite-to-martensite phase transformation. This slope change is in agreement with the results reported by Thompson[3]. In the beginning the material is in the elastic region, at the end the material is in the superelastic region and between the two regions the material in the phase transformation region.

In this study, we plotted the relation between the force and the strain. Thompson[3] probably used a wire in his experiments, so it was easy to calculate the stress (σ), using the area of the specimen. In our case, since the shape of the file is very complex, it is impossible to calculate the stress with any accuracy. However, since the stress is proportional to the force (σ = F/A), the shape of the curve is the same.

Thompson[3] mentioned that the preparation of the root canal promotes the martensite transformation by stress of NiTi alloy instruments. The stress level at which the phase transformation happens is not mentioned by the authors, and was found by us to be different for each instrument taper. This is an important information for future studies and for clinical practice.

According to Schäfer et al,[4] the cross section is the main factor that affects the bending tests. This is reasonable, since that a larger area implies a larger volume of metal at the core of the instrument. In the present study, a factor that influenced the maximum force to bend the instruments to 45º was the taper. The taper has the same influence as the cross section, for the same reason. If the tip diameter is kept constant, a larger taper will promote less flexibility, as was observed in the tests.

According to Miyai et al[5] and Hayashi et al,[6] the instrument flexibility is influenced by a phase transformation. The R-phase or rhombohedral has a large memory form effect and the Young modulus is lower than that of austenite, so an instrument that goes through a martensitic transformation will be more flexible.

Yahata et al[7] used the same scheme proposed by Miyai et al[5] to study the flexibility of annealed samples. However, differently from this study, the authors did not measure the average force for phase transformation when the instrument is submitted to stress.

Other values found in this study were compatible with NiTi alloys. Lopes et al[8] found average values of 345 HV in NiTi instruments (Files NiTi-flex). Serene et al[9] found values between 303 and 362 HV for the microhardness of NiTi alloys used in the manufacture of endodontic instruments. The average value found in the the present work was 289 HV. This value is consistent with others from the literature.

In this work it was observed that the manufacturing process did not change the Vickers microhardness, probably because of the thermal treatment, that could be lower than the temperature of recrystalization proposed by Kuhn and Jordan.[10]

According to the manufacturer,[11] the absence of other metal at the fixation cables avoids galvanic corrosion. The instrument is really formed by only one piece. However, galvanic corrosion should not be an important problem because of the low life in cycle at the clinic. It will be important only if the instrument remains in stock for a long period of time in adverse conditions.

According to Kim et al,[12] the TF® instruments present a significant resistance to fracture by rotating-bending fatigue when compared with others

instruments manufactured by the machining process, corroborating the results obtained by Gambarini et al[13] and Larsen et al.[14] This can be explained by the fact that machining produces perpendicular defects that favor nucleation and propagation of cracks.

Even presenting good results in flexion-bending fatigue tests, the TF® files should have a better surface finishing, that would improve the clinical performance concerning durability in relation to the fracture. The surface morphology found at this work was very similar to that found by Kim et al.[12] Despite the eletropolishing, the surface is not completely flat and has machining marks from the manufacturing process. This observation corroborates the results of that study.

Conclusions

Based on the results we concluded that:

a) the dimensions of the TF® files meet the ANSI/ADA standard number 28 recommendations;

b) the files present many defects from the manufacturing process;

c) the instrument flexibility decreased with increasing taper;

d) the phase transformation induced by stress average forces to the TF® files of taper 0.04; 0.06 and 0.08 mm/mm where 100 gf, 150 gf and 250 gf, respectively, and

e) the TF® Vickers microhardness values were similar to those of NiTi rotary instruments manufactured by the machining process.

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