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Influence of gold and blue heat treatments on mechanical properties and phase transformation behavior of flat-designed rotary instruments

Influência dos tratamentos térmicos gold e blue nas propriedades mecânicas e no comportamento de transformação de fase de instrumentos rotatórios com design flat

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ABSTRACT

Objective: To evaluate the mechanical properties and austenite phase transformation temperature of flat-designed rotary instruments manufactured with gold and blue heat treatments. **Material and Methods:** A total of 60 rotary instruments with a flat design 25.04 with gold (Flat File 25.04 – MK Life, Brazil) and blue (Prototype 25.04 - MK Life, Brazil) heat treatments were used for cyclic fatigue, torsional fatigue, and bending tests (n=30). The cyclic fatigue test was conducted in an artificial canal with a curvature of 60° and a radius of 5 mm (n=10). The torsion test was conducted to evaluate the torque (N.cm) and angular deflection (°) required for fracture (n=10). Finally, the 60° bending test evaluated the force (g.f) required to flexion the instruments at the 5 mm tip (n=10). Differential Scanning Calorimetry (DSC) analysis was performed to establish the initial (Ai) and final (Af) austenitic transformation temperatures. The data were statistically analyzed using the Kolmogorov-Smirnov test for normality and the unpaired t-test, with a significance level of 5%. **Results:** The cyclic fatigue test showed that Flat 25.04 gold instruments had significantly greater cyclic fatigue resistance ($p<0.05$). The torsion test revealed that Flat 25.04 gold instruments exhibited lower maximum torque (N.cm) and greater angular deflection (°) ($P<0.05$). The bending test showed that the gold instrument required less force ($P<0.05$). DSC analysis demonstrated that the gold heat treatment had a higher Af temperature (42.8 °C) compared to the blue treatment (32.2 °C). **Conclusion:** The different heat treatments impacted in phase transformation temperatures and on the mechanical properties of the instruments.

KEYWORDS

Cyclic fatigue; Endodontics; Flat design; Nickel-Titanium; Torsional fatigue.

RESUMO

Objetivo: Avaliar as propriedades mecânicas e a temperatura de transformação de fase austenita em instrumentos com design flat confeccionados com tratamento térmico gold e blue. **Material e Métodos:** Foram utilizados 60 instrumentos rotatórios com design flat 25.04 com tratamento térmico gold (Flat File 25.04 – Mk Life, Brasil) e blue (Protótipo 25.04 – Mk Life Brasil) para avaliar a resistência a fadiga cíclica, torcional e flexional (n=30). O teste de fadiga cíclica foi realizado em um canal artificial com 60° de curvatura e 5 mm de raio (n=10). O teste de torção avaliou o torque máximo (N.cm) e deflexão angular (°) (n=10). O teste de flexão foi realizado para avaliar a força necessária para flexionar os instrumentos a uma curvatura de 60° (n=10). O teste de Escaneamento Diferencial de Calorimetria (EDC) avaliou a temperatura de transformação austenita inicial (Ai) e final (Af).

Os dados obtidos foram analisados estatisticamente empregando o teste de Kolmogorov-smirnov e teste t não pareado, com nível de significância de 5%. **Resultados:** O teste de fadiga cíclica demonstrou que o instrumento flat 25.04 gold apresentou maior resistência à fadiga cíclica (P<0,05). O teste de torção demonstrou que o instrumento flat 25.04 gold apresentou menor torque e maior deflexão angular (P<0,05). O teste de flexão demonstrou que o instrumento flat 25.04 gold apresentou menor força à flexão de 60° (P<0,05). O teste de EDC demonstrou que o tratamento gold (42,8 °C) apresentou uma temperatura Af maior que o tratamento blue (32,2 °C). **Conclusão:** Os tratamentos térmicos impactaram na temperatura de transformação e nas propriedades mecânicas dos instrumentos avaliados.

PALAVRAS-CHAVE

Fadiga cíclica; Endodontia; Design flat; Níquel-Titânio; Fadiga torcional.

INTRODUCTION

Nickel-Titanium (NiTi) mechanized instruments have been widely used for preparing curved root canals due to their high flexibility, providing safety and low risk of instrumentation errors or instrument fracture [1,2]. However, instrument fracture continues to be a concern for clinicians [2].

The instruments separation during use can occur due to two causes: cyclic and torsional fatigue [3,4]. Cyclic fatigue occurs when instruments are rotating inside the curved root canal and are subjected to tensile and contraction forces at their maximum flexion point, which can lead to rupture of the metallic alloy [3,4]. Torsional fatigue occurs when the instrument's tip becomes trapped in the dentin walls while the instrument continues its rotational movement, which can lead to plastic deformation and/or instrument rupture [3,4]. Therefore, manufacturers have developed several modifications to optimize their mechanical properties, such as: new instrument designs, manufacturing processes, new kinematics, and different thermal treatments of the NiTi alloy [1,2,4-7].

The thermal treatment of NiTi provides a better arrangement of the crystalline structure of the metallic alloy, which favors the appearance of the R or martensitic phase, phases responsible for the super flexibility of the alloy and, consequently, better mechanical properties of the instruments [1,2,7] Aditionally, these treatments tend to modify the alloy transformation temperature, altering the presence of these different phases in response to the temperature of the root canal [1,7]. Therefore, it is essential that the endodontist knows the characteristics of the instruments and select them according to the anatomy to be prepared, enabling safety and effectiveness during root canal preparation [1,8,9].

Gambarini et al. [10] proposed a new design concept for instruments known as “flat”. This design involves an instrument with one of its sides flat and without a cutting surface, aiming to reduce friction with the canal walls, reduce the volume of metal and decrease debris accumulation inside the root canals. According to the authors, this characteristic provides a significant improvement in resistance to cyclic fatigue.

Some rotary systems were launched on the world market driven by this innovative design, even without any other robust scientific evidence at that time, varying with the preparation sequence, types of heat treatments, etc. Silva et al. [11] and Jeong et al. [12] demonstrated that the flat design did not provide improvements in resistance to cyclic and torsional fatigue compared to S cross-section instruments. Additionally, Silva et al. [11] demonstrated that the flat design provides similar efficiency in removing debris. Therefore, although there are few studies regarding the impact of the flat design on the mechanical properties and preparation of root canals, it is possible to state that the flat design does not provide improvements in mechanical properties compared to the conventional S cross-section.

Although the flat design cross-section does not present advantages in the mechanical properties of NiTi rotary instruments, the use of different types of heat treatments is an industrial option widely used for this purpose [7,8]. There is no study that has evaluated the mechanical properties of instruments with a flat design made with different heat treatments. The objective of this study was to compare the mechanical properties of two flat-design instruments made with gold and blue heat treatments and the NiTi alloy transformation temperatures using Differential Calorimetry Scanning. The null hypotheses of this study were was: thermal treatments do not impact the mechanical properties and the phase transformation behavior of instruments with a flat design.

MATERIAL AND METHODS

To perform mechanical tests, a sample size calculation was conducted using the G*Power v3.1 for Mac software (Heinrich Heine University Düsseldorf (HHU)) by selecting the Wilcoxon-Mann-Whitney test from the t-test family. An alpha type error of 0.05, a beta power of 0.95, and an N2/N1 ratio of 1 were also stipulated. The effect size of the sample was 1.65, and the actual power was 0.965. A total of 10 specimens per group was indicated as the ideal sample size, based on the article by Alcalde et al. [13]. Prior to the mechanical tests, all instruments were inspected for possible defects or deformities under a stereomicroscope (Stemi 2000C; Carls Zeiss, Jena, Germany) with 16X magnification.

For the mechanical tests in this study, a total of 60 rotary instruments 25.04 with a flat design, manufactured with blue and gold thermal treatment (n=30 each), were used. The instruments used in the gold treatment group are commercially available in Brazil from the company MK Life (Porto Alegre, Rio Grande do Sul, Brazil), under the brand name Flat File. Conversely, the instruments with blue thermal treatment are prototypes that differ from the Flat File instruments solely in the thermal treatment applied (Figure 1).

Cyclic fatigue test

A total of 20 rotary instruments 25.04 with a flat design, gold and blue (n=10) were used. The cyclic fatigue test was conducted using an apparatus with an artificial stainless-steel canal with a 60° curvature and a 5 mm radius under conditions simulating body temperature (36° ± 1 °C), as described by Klymus et al. [14]. The instruments were activated using a VDW Silver Reciproc electric motor (VDW, Munich, Germany) and operated in a rotary motion at 500 rotations per minute (RPM) and 2 N.cm of torque. During the test, the time until instrument fracture was measured using a digital stopwatch and confirmed by video recording simultaneously with the test. After measuring the time to fracture, the number of cycles was calculated using the following formula:

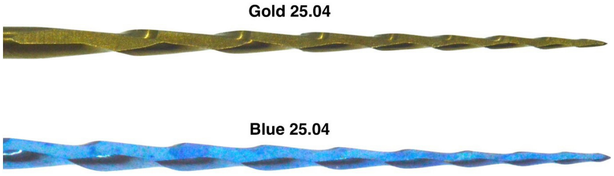

Figure 1 - Representative images of Flat 25.04 instruments with gold and blue thermal treatments.

$$\frac{time\ to\ fracture\ (in\ seconds)\ \times\ speed\ (rotations\ per\ minute)}{60} \quad (1)$$

Torsion and bending test

For torsion test, 20 rotary instruments 25.04 with a flat design, manufactured with blue and gold thermal treatment (n=10 each), were used. The torsion tests were performed according to ISO 3630-1 specification [15] and under conditions simulating body temperature (36° ± 1 °C), as previously described by Osaki et al. [16]. The torsion test evaluated the torque (N.cm) and angular deflection (°) required to fracture the instruments.

Prior to the tests, all instruments had their shafts removed to allow fixation in the torsion machine. The ends of the instruments were then fixed in screwable mandrels, with the first 3 mm of the instrument tip fixed in a mandrel coupled to a torque cell and the other end fixed in a mandrel connected to a reversible gear motor. A clockwise rotation at a speed of 2 rotations per minute (RPM) was applied to all groups. Simultaneously with the motor rotation, the software provided the torque (N.cm) and angular deflection values required for instrument fracture.

The bending test was performed using the same torsion equipment under conditions simulating body temperature (36° ± 1 °C), following an adaptation employed by Alcalde et al. [13]. A total of 20 instruments (n=10 each) were used, with 5 mm of the instrument tips fixed in a force cell and bent to 60°. The software then provided the necessary force in gram-force (g.f).

Evaluation of Instrument Surfaces Using Scanning Electron Microscopy (SEM)

This step aimed to evaluate the topographical characteristics of the fractured surfaces of the instruments subjected to torsion and cyclic fatigue tests. All instruments were examined using a scanning electron microscope (SEM) (JSM-T220A, Jeol, Tokyo, Japan) to assess the topographical characteristics of the fractured surfaces with a magnification of 35x and 500x.

Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry was employed to determine the initial austenite transformation temperature (Ai) and final austenite transformation temperature (Af) of the different thermal treatments applied. The DSC analysis was conducted according to the 2004 ASTM [17] guidelines, using five new instruments. A fragment of 3 to 5 mm, weighing 7 to 12 mg, was extracted from the active part of the instrument and treated with a mixture of 25% hydrofluoric acid, 45% nitric acid, and 30% distilled water for two minutes. The specimens were subsequently cleaned with distilled water and mounted on an aluminum support, with an empty support serving as the control. All analyses were performed using the Differential Scanning Calorimetry Equipment (Mettler Toledo, Tamboré - Barueri - SP, Brazil), connected to a Samsung desktop computer (Samsung Digital City, Maetan-dong, Yeongtong). The STARe software (Mettler Toledo) was used, and the DSC graphs were extracted.

The thermal cycles lasted 45 minutes and included the following steps: isothermal retention at 25 °C for 5 minutes, heating to 150 °C (at a rate of 10 °C/min), an isothermal hold for 2 minutes, cooling to -30 °C (at a rate of 10 °C/min), an isothermal hold for 2 minutes, heating to 150 °C (at a rate of 10 °C/min), and finally, an isothermal hold for 2 minutes, followed by cooling to 25 °C. The phase transformation temperature results were analyzed using the Netzsch Proteus Thermal Analysis software (Netzsch-Garatebau GmbH). The test was performed twice for each group to confirm the results.

Statistical analysis

The data obtained from the cyclic fatigue, torsional, and bending tests were analyzed using the Kolmogorov-Smirnov test to evaluate the normal distribution. The data were then assessed using the unpaired Student's t-test, with a significance level of 5%.

RESULTS

Cyclic fatigue, torsional, and bending tests

The mean values and standard deviations for the cyclic fatigue, torsional, and bending tests are shown in Table I. The results demonstrated that the Flat 25.04 gold instruments had significantly greater time and NCF compared to the Flat 25.04 blue instruments ($P<0.05$). The torsion test showed that the Flat 25.04 gold instruments exhibited lower maximum torque (N.cm) and greater angular deflection (°) compared to the Flat 25.04 blue instruments ($P<0.05$). Finally, the bending test demonstrated that the gold instrument required less force compared to the blue instrument ($P>0.05$).

Scanning Electron Microscopy (SEM)

The SEM analysis of the fractured surfaces of all instruments subjected to cyclic fatigue and torsional tests demonstrated typical characteristics of cyclic fatigue and torsional failure. After the cyclic fatigue test, typical patterns of microcavities and topographical features of ductile fracture were observed (Figure 2). Regarding the torsional test, the instruments exhibited concentric abrasion marks indicative of stress and torsional failure. Fibrous marks were observed at the center of rotation, confirming torsional failure (Figure 3).

DSC analysis

The DSC graphs demonstrated different phase transformation temperatures for the gold and blue 25.04 instruments. The initial (Ai) and final (Af) austenite transformation temperatures are presented in the heating curve (left to right). The blue-treated instrument exhibited Ai and Af

Table I - Mean and standard deviation of cyclic fatigue, torsional fatigue, and flexural fatigue tests for Flat 25.04 gold and blue instruments

	Cyclic Fatigue		Torsional Fatigue		Bending 60°
	Time	Cycles to fracture (NCF)	Torque (N.cm)	Angular deflection (°)	Force (g.f)
Flat 25.04 gold	255.3 ± 30.73[a]	2119 ± 210.1[a]	0.81 ± 0.183[a]	398.1 ± 30.2[a]	139.6 ± 17.8[a]
Flat 25.04 blue	199.9 ± 18.28[b]	1659 ± 151.7[b]	1.2 ± 0.125[b]	338.8 ± 34.9[b]	178.1 ± 21.1[b]

Different lowercase letter in columns indicate significant difference among the instruments ($P<0.05$).

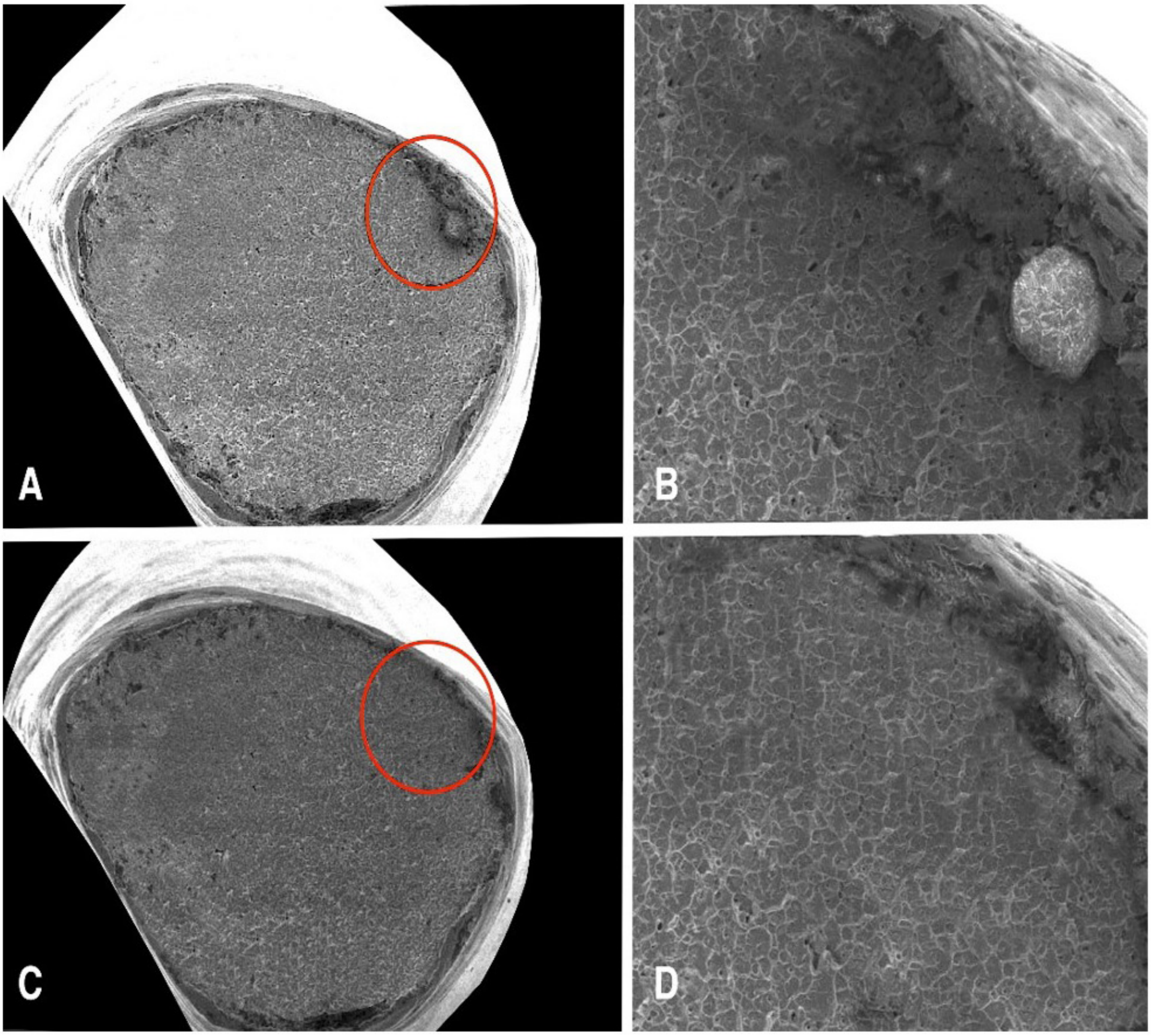

Figure 2 - Representative Scanning Electron Microscopy (SEM) images of Flat instruments with gold and blue treatments after cyclic fatigue. Images A and C show the gold and blue instruments with 35x magnification, respectively. Images B and D show representative images of the fractured surfaces with 500x magnification for gold and blue, respectively. The red circle highlights the area selected for 500x magnification.

temperatures of 23.1 °C and 32.2 °C, respectively, while the gold-treated instrument showed Ai and Af temperatures of 22.4 °C and 42.8 °C, respectively (Figure 4). Considering the temperature at which the mechanical tests were conducted (36 °C), the blue-treated instrument would undergo complete austenitic transformation, while the gold-treated instrument would exhibit a combination of austenite with R-phase.

DISCUSSION

Comparing the mechanical properties of NiTi rotary instruments available on the market is crucial for understanding the initial behavior of these instruments during root canal preparation [2]. However, the various design characteristics (taper, cross-section, core diameter), surface treatments, and thermal treatments of NiTi make this process challenging, as they directly influence the mechanical properties and must be considered in studies [9,13].

Previous studies have evaluated the cyclic fatigue and torsional resistance of rotary and reciprocating instruments with identical designs and different thermal treatments [18-22]. The authors demonstrated that thermal treatments have a significant impact on mechanical properties, as they alter the amount of R-phase and martensite in the NiTi alloy structure, making it more flexible compared to alloys

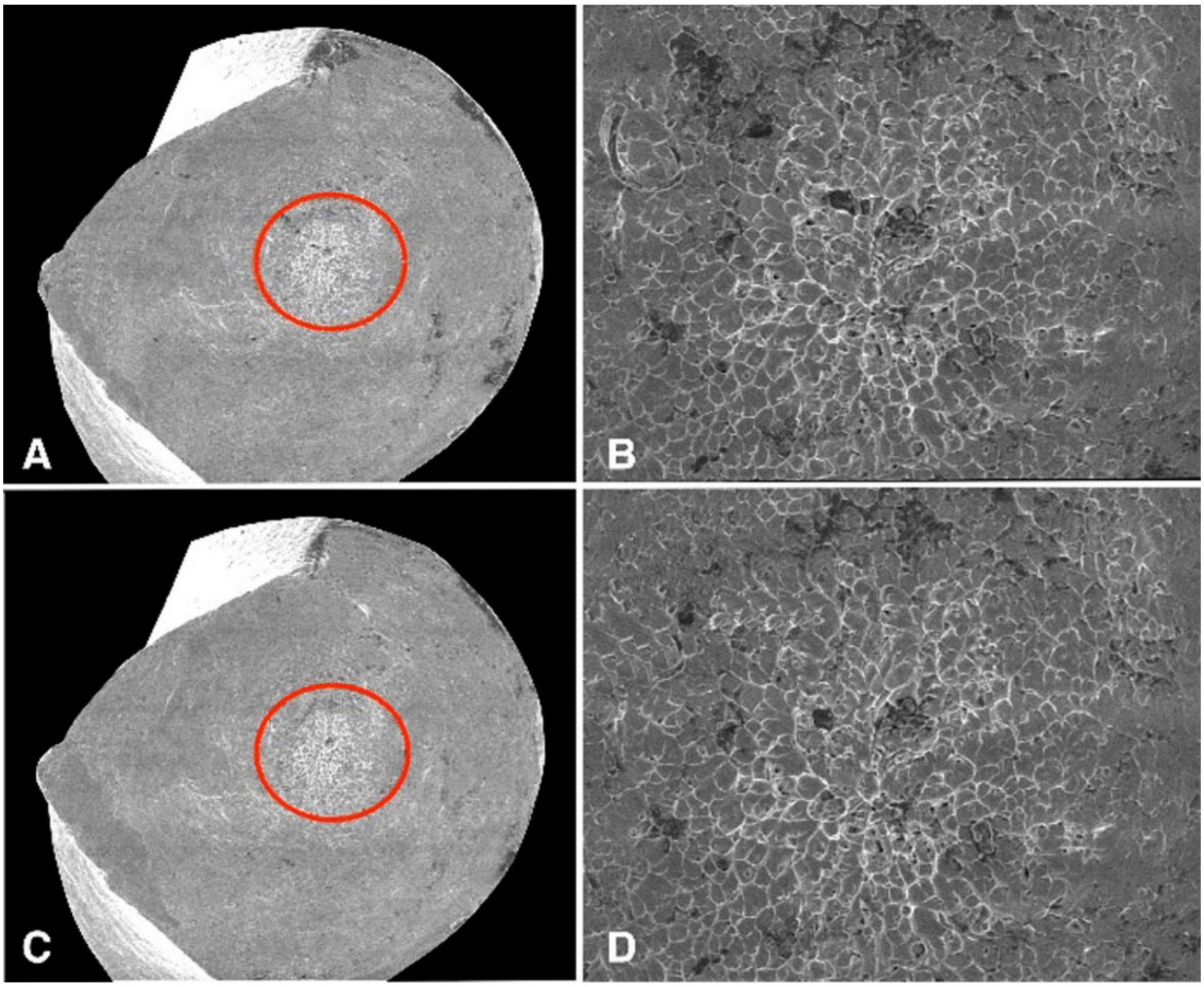

Figure 3 - Representative Scanning Electron Microscopy (SEM) images of Flat instruments with gold and blue treatments after torsional fatigue. Images A and C show the gold and blue instruments with 35x magnification, respectively. Images B and D show representative images of the fractured surfaces with 500x magnification for gold and blue, respectively. The red circle highlights the area selected for 500x magnification.

with predominant austenite phase [7,19]. Furthermore, this increased flexibility tends to provide more centered canal preparations in canals with sharp curvatures [1,7,23].

There is no consensus in the literature regarding which thermal treatment is considered ideal, as this characteristic should be evaluated according to the type of root canal anatomy [23,24]. Furthermore, no studies have assessed the mechanical properties of flat-designed instruments with different thermal treatments. The results of this study demonstrated that the Flat 25.04 gold instruments exhibited a greater time and higher number of cycles to fracture compared to the Flat 25.04 blue instruments. Additionally, the gold treatment provided lower maximum torque and greater angular deflection to fracture compared to the blue treatment. Moreover, it required less force for bending compared to the blue treatment. Therefore, the initial hypothesis was rejected.

Considering that the instruments have identical designs, the likely and sole explanation for the differences observed in this study is the varying percentage of R-phase and martensite between the gold and blue treatments. Moreira et al. [21] evaluated the mechanical properties of Profile 25.06 instruments made from conventional NiTi alloy, with blue and gold thermal treatments. The authors demonstrated that both gold and blue thermal treatments provided greater cyclic fatigue resistance compared to instruments made from conventional NiTi. Additionally, the gold treatment exhibited significantly higher cyclic fatigue resistance (both in terms of time and number of cycles) and lower bending resistance compared to the blue treatment. These findings support the results of our study.

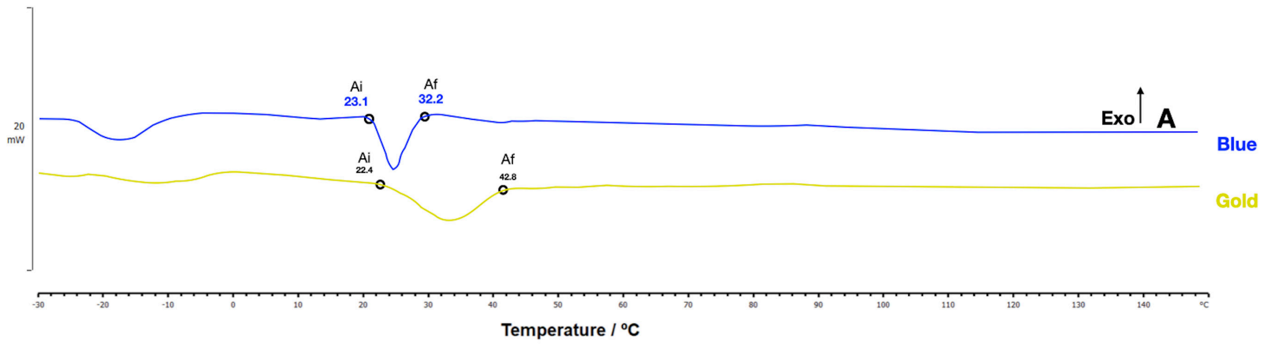

Figure 4 - Representative image of the Differential Scanning Calorimetry (DSC) graph showing the transformation temperatures of initial austenite (Ai) and final austenite (Af). The lines represent the heating curves identifying Ai and Af (left to right).

Although no studies have assessed the torsional resistance of instruments with identical designs and blue and gold thermal treatments, several previous studies have shown differences in the presence of R-phase and martensite between the two treatments [1,7,22]. Therefore, the torsional results presented by the Flat 25.04 gold instrument indicate greater flexibility compared to the blue, corroborating the results obtained in the cyclic fatigue and bending tests of this study.

The SEM analysis showed the typical features of cyclic and torsional fatigue for both thermal treatments. After the cyclic fatigue test, the instruments showed areas of crack initiation and overload zones, with numerous dimples spread across the fractured surface. After the torsional test, the fragments showed concentric abrasion marks and fibrous dimples at the center of rotation, as previously reported [8,13].

Differential Scanning Calorimetry (DSC) is a fundamental analysis for understanding the transformation temperature of NiTi alloy thermal treatments and has been previously explored in various studies [1,7,23]. Knowledge of the transformation temperature allows for understanding whether the NiTi alloy will exhibit an austenitic or martensitic behavior during root canal preparation. Additionally, it is a complementary analysis to the mechanical test results. In this study, the DSC analysis demonstrated that the final transformation temperature (Af) for the gold treatment is higher than that for the blue treatment, thus, the second hypothesis of this study was rejected.

The different Af transformation temperatures between the gold (42.8 °C) and blue (32.2 °C) treatments significantly impacted the results of this study, as all tests were conducted in an environment simulating body temperature. Therefore, the blue treatment exhibited complete austenitic transformation at 36 °C, making it less flexible compared to the gold treatment. These results complement and justify those found in the mechanical tests. Furthermore, the findings of this study are consistent with the results of Moreira et al. [21].

Laboratory mechanical tests of cyclic fatigue, torsion, and bending are used to estimate how the instruments would behave during the preparation of curved and/or constricted canals. Additionally, the bending test provides an estimate of which type of instrument is likely to exert greater force against the walls of the root canals and, consequently, a higher chance of deviations [21,23]. However, mechanical tests do not necessarily directly translate into greater safety or efficacy during root canal treatments, which requires additional studies, such as evaluations in ex vivo models or clinical studies to confirm our results.

CONCLUSION

Different thermal treatments significantly impact the resistance to cyclic fatigue, torsion, and bending of the instruments, with the gold treatment exhibiting greater flexibility. The DSC analysis demonstrated that the gold treatment had a higher final transformation temperature (Af) compared to blue.

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Author's Contributions

ARTV: Writing – Original Draft Preparation. GFS: Writing – Review & Editing. PAAS: Investigation. RRV: Investigation. MAHD: Writing – Review & Editing. TOL: Writing – Original Draft Preparation. MCGO: Writing – Original Draft Preparation. MPA: Supervision.

Conflict of Interest

No conflicts of interest declared concerning the publication of this article.

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Regulatory Statement

Ethics approval was not required.

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