

Influence of heat treatment on color and flexibility of nickel-titanium endodontic instruments

Influência do tratamento térmico na cor e flexibilidade de instrumentos endodônticos de níquel-titânio

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ABSTRACT

Objective: The aim of this study was to evaluate and compare the effect of heat treatment at different temperatures on the color and flexibility of nickel-titanium endodontic instruments. **Methods:** Thirty-six nickel-titanium K-file endodontic instruments were divided into six groups and submitted to heat treatment at several temperatures between 450°C and 750°C. The color was visually observed. Cantilever-bending tests were performed to measure the flexibility of the instruments. The data were statistically evaluated by one-way analysis of variance and Tukey post hoc comparisons. **Results:** It was observed that the color becomes darker as the heating temperature increases. The flexibility is significantly higher after the heat treatment ($P < 0.05$). Instruments treated at 650°C and 750°C had higher flexibility than those treated at lower temperatures (450°C, 500°C and 550°C) ($P < 0.05$). **Conclusion:** Heat treatment has a significant effect on the surface color and flexibility of nickel-titanium endodontic instruments. The highest flexibility was observed in instruments heated at 650°C and 750°C.

Indexing terms: Dental instruments. Heat treatment. Pliability.

RESUMO

Objetivo: O objetivo deste estudo foi avaliar e comparar o efeito do tratamento térmico realizado em diferentes temperaturas sobre a cor e a flexibilidade dos instrumentos endodônticos de níquel-titânio. **Métodos:** Trinta e seis instrumentos endodônticos de níquel-titânio tipo K foram divididos em seis grupos e submetidos ao tratamento térmico com temperaturas variando entre 450°C e 750°C. A cor dos instrumentos após tratamento foi visualmente observada. Testes de flexão em 45° foram realizados para medir a flexibilidade dos instrumentos. Os dados foram avaliados estatisticamente através de análise de variância (ANOVA) com teste post hoc de Tukey. **Resultados:** Observou-se que os instrumentos apresentaram cor mais escura à medida que a temperatura de aquecimento foi aumentada. A flexibilidade é significativamente maior em instrumentos submetidos ao tratamento térmico ($P < 0,05$). Os instrumentos tratados a 650°C e 750°C tiveram maior flexibilidade do que aqueles tratados em baixas temperaturas (450°C, 500°C e 550°C) ($P < 0,05$). **Conclusão:** O tratamento térmico afeta significativamente a cor apresentada pela superfície dos instrumentos endodônticos de níquel-titânio e sua flexibilidade. A maior flexibilidade foi observada em instrumentos tratados nas temperaturas de 650°C e 750°C.

Termos de indexação: Instrumentos odontológico. Tratamento térmico. Maleabilidade.

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INTRODUCTION

The use of nickel-titanium (NiTi) alloy in the manufacture of endodontic instruments led to a significant improvement in the quality of dental root canal treatment by reducing the number of sessions and decreasing errors during mechanical preparation [1-4]. NiTi files have greater flexibility than traditional stainless steel instruments, allowing a more precise and refined instrumentation of the root canal [5-8].

Despite advances in manufacturing process and design of the NiTi instrument, preparation of root canals with severe curvature still presents a challenge for the endodontist [9,10]. In order to improve flexibility and fatigue resistance of NiTi instruments, different approaches have been proposed in recent years. Some commercially available NiTi instruments were submitted to thermomechanical treatment and received designations as M-Wire (Dentsply Sirona, Ballaigues, Switzerland), CM Wire (Coltene/Whaledent, Altstätten, Switzerland) and R-Phase alloy (SybronEndo, Orange, CA, USA), among others [11,12].

The heat treatment decreases the residual stress in the instruments produced by the machining process, controls the presence and distribution of nickel-rich precipitates, and changes the parameters of the phase transformation of the NiTi alloy, such as the temperature and load required for the austenite-martensite transformation to occur [13,14].

In addition to improving the mechanical properties, heat treatment promotes changes in the superficial layer of the NiTi alloy, leading to color changes [15]. The superficial layer is composed mainly of titanium oxide (TiO) [16]. The temperature, time and rate of heating and cooling used in the heat treatment have a direct influence on the characteristics of the titanium oxide layer, resulting in variations in color [15,16]. Vortex Blue (Dentsply Sirona, Ballaigues, Switzerland), Protaper Gold (Dentsply Sirona, Ballaigues, Switzerland) and Reciproc Blue (VDW, Munich, Germany) are commercially available examples of NiTi files that have different colors.

Literature results show that the heat treatments change the mechanical properties of NiTi alloy [1]. However little is known of the influence of heat treatments on some characteristics of NiTi endodontic instruments. The purpose of this work was to study the influence of heat treatments on the surface color and flexibility of NiTi endodontic instruments.

METHODS

Thirty-six NiTi K-file (VDW, Munich, Germany), size 40 and 25 mm long, instruments were used in this study. They were divided into six groups as follows: one group without heat treatment and five groups submitted to heat treatments at different temperatures.

The samples were placed in a ceramic crucible, heat treated in a furnace (NBD-O1200, Nobody Material Science and Technology CO, Xin Cun, Henan, China) at constant temperature for 10 min, then quenched in water at room temperature. The temperatures chosen for performing the heat treatment were 450°C, 500°C, 550°C, 650°C and 750°C. The heat treatment parameters were chosen after preliminary tests.

The instrument flexibility was measured by a bending test. The apparatus shown in Figure 1 was used to reduce the friction between the sample and the test device. The bending test followed the ANSI/ADA no 28 and ISO 3630/1 specifications [18,19]. A universal testing machine (DL 10.000, Emic, São José dos Pinhais, PR, Brazil) was used. The loading point was 3.0 mm from the instrument

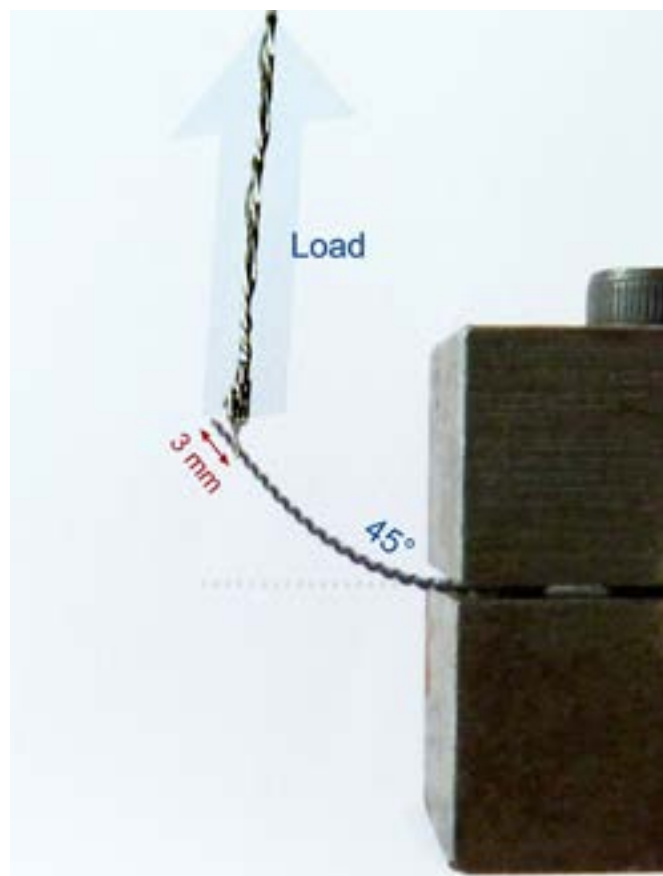


Figure 1. Set up for a cantilever bending test.

tip. The apparatus was set to stop the angular deflection at 45°. A load cell of 20 N was used and the loading speed was 15 mm/min.

Mean scores and standard deviations were calculated for each group. The data were evaluated using one-way analysis of variance and Tukey post hoc comparison (SPSS v20.0, SPSS Inc, Chicago, IL) for differences between groups at a 95% confidence level ($P < 0.05$).

RESULTS

The color of NiTi instruments varied according to the temperature used in each heat treatment. Figure 2 illustrates the color change on the surface after heat treatment with temperature range between 450°C and 750°C. It was observed that the color gradually becomes darker as the temperature increases. The instrument without heat treatment presented a silver white surface color. When the temperature was increased to 450°C, the surface color became golden orange. The color on the surface of the instruments was royal blue at 500°C. At temperature of 550°C, a slightly light green color was

observed on the surface of the instruments, followed by dark grey on 650°C samples. Finally, at 750°C, the instruments presented a brownish black color surface.

Figure 3 shows the typical load curve of a sample. The results of the bending test are shown in table 1. The

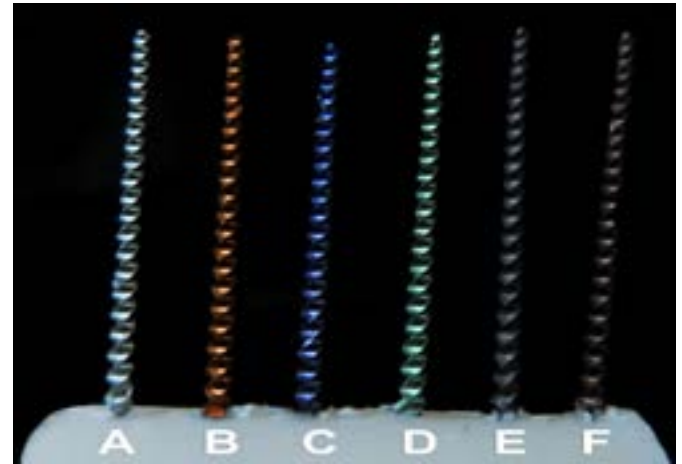


Figure 2. NiTi endodontic instruments after heat treatment at different temperatures. (A) Without heat treatment; (B) 450°C; (C) 500°C; (D) 550°C; (E) 650°C; (F) 750°C.

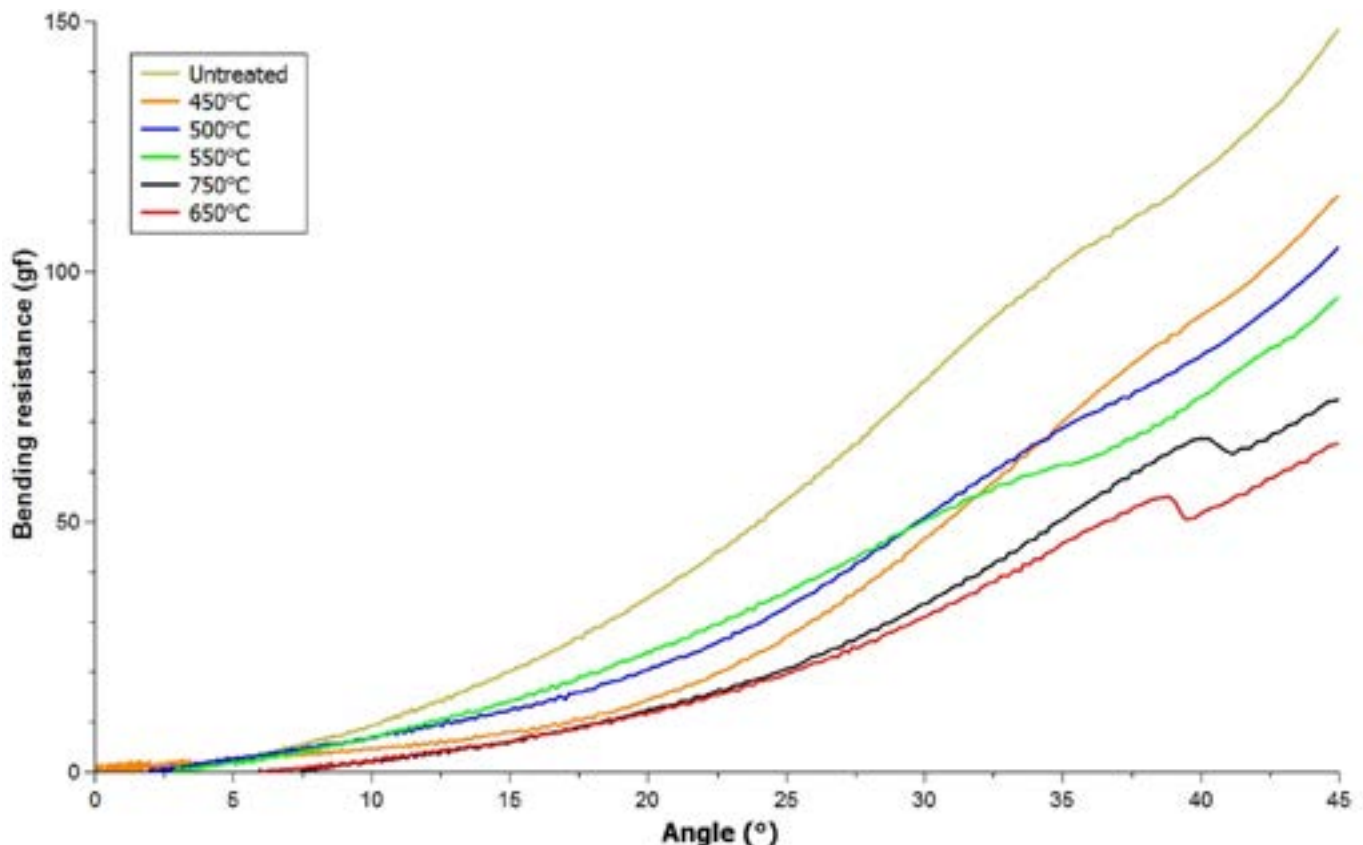


Figure 3. Bending resistance-angle curves obtained from a bending resistance test.

Table 1. Force to bend instruments up to 45 degrees (mean \pm standard deviation) and color after heat treatment.

Group	Bending resistance (gf)	Color
Untreated Group	114.15 \pm 6.83 ^a	silver white
450°C Group	88.65 \pm 4.94 ^b	golden orange
500°C Group	82.50 \pm 8.25 ^b	royal blue
550°C Group	70.72 \pm 6.37 ^d	light green
650°C Group	57.78 \pm 3.92 ^c	dark grey
750°C Group	65.54 \pm 7.80 ^c	brownish black

^{a,b,c,d}Different superscript letters indicate statistical differences between groups ($P < 0.05$).

flexibility of the samples increased significantly for all heat treatments ($P < 0.05$).

Comparing the influence of the heat treatment temperature on sample flexibility, it was observed that samples treated at 450°C and 500°C showed no significant difference in bending resistance ($P > 0.05$). Samples treated at 550°C showed higher flexibility than samples treated at 450°C and 500°C ($P < 0.05$), but lower flexibility than samples treated at 650°C and 750°C ($P < 0.05$). No statistically significant difference was found between samples treated at 650°C and 750°C ($P > 0.05$).

DISCUSSION

In the present study, the color and flexibility of NiTi endodontic instruments was changed by a heat treatment. Several studies have investigated the effects of heat treatment on NiTi alloys [1,2], but few studies [2,3] have evaluated the influence of heat treatment on NiTi endodontic instruments. Conventional NiTi endodontic files from the same manufacturer, with identical sizes, shapes and lot numbers were used in this study. This procedure eliminated possible factors that could influence the results, such as differences in thermomechanical treatment, electrochemical polishing, alloy composition and cross section.

The color change on NiTi endodontic instruments after heat treatment is a product of variations in the titanium oxide layer locate in the external surface of the instrument [20,21]. Studies have shown that the thickness of the titanium oxide layer varies according to the temperature used in the treatment, which at higher temperatures the formed surface layer is thicker than at low temperatures [21-23], leading to darker colors [22,23]. These studies are in agreement with the results observed

in this present study, which at lower temperatures of heat treatment (450-550°C) the predominance of lighter colors such as golden orange, blue and light green were observed, whereas at higher temperatures (650-750°C), the NiTi alloy presented dark surfaces, such as dark grey and brownish black.

Several studies have shown that the color presented by titanium base alloy does not depend exclusively on the thickness of the titanium oxide layer formed during the heat treatment but also on the type and concentration of the oxide formed on the alloy surface [22,23]. It was observed the preferential formation of certain types of titanium oxides, such as TiO, TiO₂ and Ti₂O₃, after heat treatment at 450°C to 750°C [15]. The analyze of type of oxide on the endodontic instruments was not objective of the present work.

The color presented by the NiTi alloy after the heat treatment may be important as indicative of the characteristics and mechanical properties expected of an endodontic instrument confectioned from that alloy. In different areas, such as aerospace and naval, the color presented by an alloy after heating is used for quality control and estimation of mechanical properties [15,16]. Similarly, in endodontics, it should be possible to estimate the mechanical properties of a NiTi endodontic instrument from its color, since it is related to the heat treatment to which it was submitted.

Several studies have demonstrated that heat treatment promotes significant improvements in some of the mechanical properties presented by NiTi files, such as fatigue resistance and flexibility [24-26]. Root canal curvature and the flexibility of the endodontic file have a direct influence on potential errors that may occur during canal preparation such as loss of working length, apical deviation and file separation. A high flexibility decreases

stress on the file and risk of fracture, since the load exerted on the instrument, when inside a root canal, is reduced [1].

In the present study, the influence of the heat treatment on the flexibility of the endodontic instrument was investigated. The endodontic instrument that received a heat treatment had higher flexibility, requiring a lower load to bend during the test. The curves obtained in the bending test (Figure 3) have a higher slope for samples that had no heat treatment when compared to the curves of instruments treated at any of the temperatures used, indicating that a higher load is required to bend the instrument. These results are in agreement with the results of other studies comparing the flexibility of instruments made from heat treated alloys with instruments made from conventional NiTi alloys [27].

The thermal treatment decreases the stresses introduced in the manufacturing process [13,29], improving the mechanical properties of the instrument. The thermal energy during heating reduces the crystalline defects in the metal structure and improves the material mechanical properties. Thus, even in samples that were treated at relatively low temperatures (450-550°C) there is a significant increase in flexibility relative to untreated samples. The samples treated at 650°C and 750°C showed greater flexibility than samples treated at lower temperatures, but samples treated at 650°C are slightly more flexible than samples treated at 750°C. This result suggests that increasing the treatment temperature improves the mechanical properties of the instrument only up to a certain point. Studies have shown [28] that heat treatments at temperatures above 800°C, cause negative effects on the mechanical properties of NiTi alloys, resulting in a loss of superelastic behavior and recrystallization of the wrought microstructure.

Commercially NiTi endodontic instruments have austenite (superelastic), martensite (heat treated alloys: M-Wire, CM Wire and R-Phase) or a mixture of the two phases. Heat treated alloys have higher ductility than the conventional NiTi alloy and a higher capacity to accommodate stresses without deformation of the [24].

Heat treatment can change the mechanical behavior of the alloy through changes in the microstructure and/or phase transformation to occur [24]. Thus, the greater flexibility presented by samples treated at 650 °C and 750 °C may occur not only by changes in the microstructure that allow stress reduction but also by the phase transformation that the samples undergo when they are subjected to a tension during the tests. As can be seen

in Figure 3, the curves representing the tests of samples treated at 650°C and 750°C show a plateau, indicating that a phase transformation occurs when the alloy is subjected to loads above a critical value.

The results of the present work show that the heat treatments of NiTi endodontic instruments change their mechanical properties. These results can help the selection of endodontic file for different dental root canal shape and improve their clinical performance. However, further studies may be necessary to investigate the chemical composition and crystalline structure of the oxide layer formed on the surface after NiTi of the heat treatment.

CONCLUSION

The results of this work showed that heat treatments improve the mechanical properties of the NiTi instruments. The surface color of the instrument depended on the temperature of the heat treatment: the color becomes darker as the heating temperature increases. Heat treated instruments were significantly more flexible than untreated instruments and samples treated at 650 °C and 750 °C were more flexible than samples treated at lower temperatures.

Collaborators

BC ALMEIDA performed laboratorial evaluations, data acquisition and writing. CN ELIAS developed the experimental design, article evaluation and writing.

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