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Effect of heat treatments on Ni-Ti endodontic files

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Abstract

Three brands of rotary nickel-titanium endodontic instruments with some comparable geometric features but with different cross-section (similar tip size and same taper of .04) were selected for this study: MTwo .04(35) (VDW GmbH, Germany), K3 .04(30) (SybronEndo, Mexico) and K3XF .04(30) (SybronEndo, Mexico). K3XF is made from novel R-phase heat-treated metal alloy while all other files were made from traditional Ni-Ti alloy. The instruments were analysed under the following conditions: i) as-received (AR), ii) heat treated at 350ºC, iii) heat treated at 400ºC. The transformation temperatures were determined by differential scanning calorimeter (DSC). Compared to conventionally K3 and MTwo files, R-phase heat treatment K3XF file showed higher transformation temperatures. These results also showed that heat treatments increase the transformation temperatures.

Keywords: NiTi shape memory alloys; endodontic files; heat treatments; transformation temperatures.

1. Introduction

NiTi rotary files offer more distinct clinical advantages with curved root canals than stainless-steel instruments, due to their higher flexibility, by virtue of their superelasticity [1].

This functional property presented by NiTi has made it possible to carry out extremely conservative shapes, better centred, with less canal transportation and therefore with more respect for the original anatomy of the tooth [2].

The two main phases (martensite and austenite) of this material are associated to the shape memory effect and superelasticity, but also an intermediate phase (R-phase) may occur. The presence of the latter phase is considered by some authors to be the main reason for increased flexibility of NiTi instruments over traditional stainless steel ones [3]. Formation of the R-phase is favoured by the presence of Ni_4Ti_3 due to the formation of stress fields around the precipitates [4]. As such, proper heat treatments that may enable Ni_4Ti_3 precipitations may improve the superelastic effect in NiTi endodontic files.

Despite the advantages of NiTi instruments, the possibility of an instrument separation is not uncommon and is a major concern during the clinical use of NiTi files. Cyclic fatigue failure is reported to occur unexpectedly without any sign of a previous permanent plastic deformation. This occurs because of repeated compressive and tensile stresses accumulated at the point of maximum flexure of an instrument rotating in a curved canal [5,6].

Possible strategies to increase the resistance to cyclic fatigue of NiTi rotary instruments include, for example, R-phase heat treatment [7-9]. This special thermal process provides instruments with greater flexibility and increased resistance to cyclic fatigue than the files manufactured from traditional NiTi alloy. Recent studies evaluated the cyclic fatigue of NiTi rotary instruments produced by this new manufacturing technique. It was reported that the post-grinding heat treatment seems not only to improve flexibility of NiTi rotary instruments, but also eliminate many drawbacks of the grinding

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process, and can provide a superior mechanical resistance.

Previous studies investigated whether the resistance to torsional stress and cyclic fatigue would be improved by the novel R-phase heat treatment by using instruments with identical geometric characteristics, like K3 and K3XF [8]. It was observed that, promoting an increase in the R-phase fraction (by lowering the temperature of the test), the number of cycles until fracture increased significantly: the fatigue life of those devices was doubled when the R-phase fraction was of nearly 50%, with the remaining being austenite [9].

K3 was first developed in 2002 and it was manufactured with a traditional grinding process, whereas K3XF was developed in 2011 by a new heat treatment process proposed by the manufacturer.

The purpose of this study was to utilize DSC to investigate the effect of heat treatments on the transformation temperatures of popular NiTi rotary files (MTwo and K3) compared to the most recent K3XF.

2. Experimental

Three brands of rotary nickel-titanium endodontic instruments with some comparable geometric features but with different cross-section (similar tip size and same taper of .04) were selected for this study: MTwo .04(35) (VDW GmbH, Germany), K3 .04(30) (SybronEndo, Mexico) and K3XF .04(30) (SybronEndo, Mexico). K3XF is made from novel R-phase heat-treated metal alloy while all other files were made from traditional Ni-Ti alloy. MTwo and K3XF files are shown in Fig. 1.

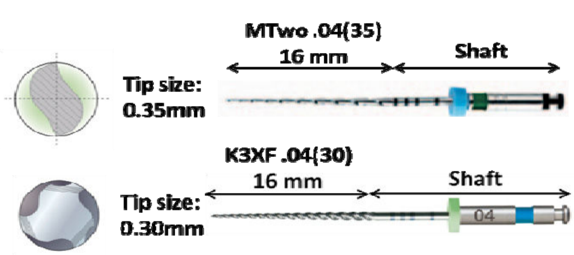


Fig. 1. Diagram of MTwo and K3XF showing the cross-section geometry.

The instruments were analysed under the following conditions: i) as-received (AR), ii) heat treated at 350ºC, iii) heat treated at 400ºC. The heat treatment (HT) was performed by holding the specimens at the aforementioned temperature for 60 min, with a heating rate of 100ºC/min, in a furnace and subsequently quenched into water.

The transformation temperatures were determined by differential scanning calorimeter (DSC) in which the shafts of the instruments were cut into three pieces weighting approximately 50 mg and placed in an aluminium crucible in the measuring chamber of a DSC apparatus (Netzsch DSC 204 F1 Phoenix). With an empty aluminium crucible placed as the reference, the chamber was filled with nitrogen gas. Gaseous nitrogen acted as coolant and the heating and cooling rate was set to 10ºC/min. The temperature range of analysis was set between -150 and 100ºC.

3. Results and Discussion

The chemical composition of the analysed files is, in average, 50.5 at.% Ni-Ti [10].

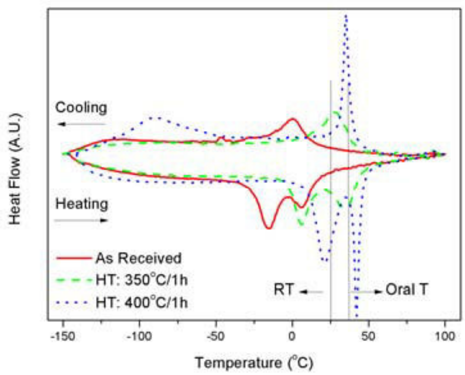
DSC curves of the shaft region of K3, K3XF and MTwo AR and HT files are shown in Figs. 2 a), b), c), respectively.

A pair of endothermic peaks can be seen on the heating curve of all the NiTi rotary files between -30 and 50ºC, even after the heat treatments. These peaks correspond to the initial transformation of martensite (B19' monoclinic structure) to R-phase (trigonal structure) at lower temperatures, followed by transformation at higher temperatures of R-phase to austenite (B2 cubic structure). This last transformation is complete, for the AR files, before 37ºC, known as the oral temperature. K3XF is the file with the highest transformation temperatures, for all the cases (AR and heat treated). Nevertheless, K3XF is also the file that shows the least influence from those heat treatments; as for the other files, the transformation temperatures increased more significantly.

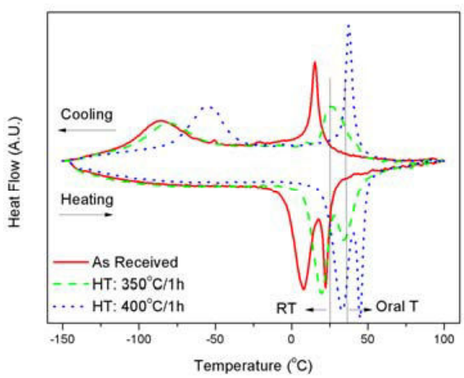
The transformation temperatures for the three as-received files and after the two heat treatments are shown on Table 1.

Table 1 shows the transformation temperatures only for the R→B2 (upon heating) and B2→R (upon cooling) transformations. The martensitic transformation temperatures were not calculated because, in most cases, the peak associated to that transformation is not clear, due to significantly spreading of this transformation up to temperatures below -150ºC. However, this peak is clear in all K3XF files and in MTwo and K3 files after the heat treatment at 400ºC for 1h. The transformation R→B19' (upon cooling) always appears following B2→R [11].

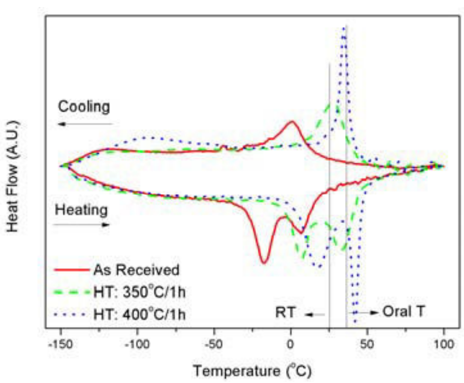
The heat treatment that shows the major difference is the one performed at 400ºC for 1h. After such heat treatment, for all files, the transformation peak corresponding to the martensitic transformation, is completed before -150ºC. Also, the transformation temperatures are significantly modified (to higher temperatures and the peaks were less spread), when compared to the AR files.



a)



b)



c)

Fig. 2. DSC curves for: K3 (a), K3XF (b) and MTwo files (c), AR, HT 350ºC/1h, HT 400ºC/1h.

The DSC results showed that all instruments in the HT condition have an A_f temperature above 37ºC, which is assumed to be the temperature at the oral environment.

A mixture of R-phase and austenite shall then be present during their clinical use at oral temperature, with the superelastic properties being dependent on the ratio austenite/R-phase during clinical application.

Table 1. Phase transformation temperatures (ºC).

		Cooling		Heating	
	HT	R_s	Rf	A_s	A_f
MTwo	AR	16.1	-17.3	-8.8	16
	350ºC/1h	40	8.5	11.5	47
	400ºC/1h	42.1	25.1	31.6	48
K3	AR	14.1	-17.4	-4.9	16.6
	350ºC/1h	41.0	5.0	7.8	46.0
	400ºC/1h	47.1	27.6	30.1	47.6
K3XF	AR	22.6	2.6	13.6	31.1
	350ºC/1h	42.6	15.1	24.6	46.1
	400ºC/1h	45.1	30.1	36.1	49.6

It has been reported that appropriate heat treatments can increase the transformation temperatures and improve the flexibility of endodontic files [12] and it has also been suggested that the heat treatments cause a decrease of the density of crystal lattice defects and contribute to changes in the phase transformation temperatures [12,13]. Compared to the AR instruments, the transformation temperatures for the HT files were shifted to higher temperatures. These observations suggest that ideal heat treatment temperatures should be below 350ºC in order to obtain transformation temperatures closer to RT. Nonetheless, the presence of both R-phase and austenite at the oral temperature should give a good mechanical behaviour during service. The influence of the heat treatments on the cycle life of the files during clinical use is currently being assessed.

4. Conclusions

The following conclusions can be drawn:

– Heat treatments increased the transformation temperatures. This raise in the transformation temperatures is more notorious as the heat treatment temperature increases;

– K3XF is the file which shows the highest transformation temperatures, thus suggesting that this file may have a previous heat treatment, which appeared not to be the case for MTwo and K3 files;

– The DSC results showed that both direct and reverse transformations are taking place in two steps B2 ⇔ R-phase ⇔ B19';

– These observations indicate that an improvement in flexibility should be expected after heat treatment, as a consequence of the presence of R-phase. In order to obtain transformation temperatures between room temperature and the oral temperature, the heat treatments for these files should be performed below 350ºC.

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