

## Effect of heat treatment on K3, K3XF and MTwo endodontic files

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**Abstract.** The purpose of this study was to evaluate the effect of heat treatment on the transformation temperatures of K3, K3XF and MTwo endodontic files. DSC was used to investigate the domains of transformation temperatures and how the transformation temperatures vary along the file. The files were analysed as received and subjected to heat treatment. X-ray diffraction analysis was also used to investigate the stress-induced martensite transformation on the files under flexion. The results show that heat treatment influences the transformation temperatures and that under flexion, the conditions and situations in which files present a stress-induced martensite transformation can be identified.

### 1 Introduction

NiTi rotary files offer more distinct clinical advantages with curved root canals than stainless-steel instruments, due to its higher flexibility, by virtue of its superelasticity [1]. The superelasticity has made it possible to carry out extremely conservative shapes, better centered, with less canal transportation and therefore with more respect of the original anatomy [2]. NiTi shape memory alloys undergo transformation from cubic austenitic to monoclinic martensite when the applied stress in the austenite phase is enough to promote the stress-induced transformation [3, 4]. This stress-induced martensitic transformation reverses spontaneously upon release of the stress and the material then returns to its original shape and size [4]. Furthermore, the rhombohedral R-phase formation, thermoelastic, often precedes the martensitic transformation under certain conditions and is considered to be the main reason for increased flexibility of NiTi instruments over traditional stainless steel ones [5].

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 [6, 7].

Possible strategies to increase the resistance to cyclic fatigue of NiTi rotary instruments include, for example, R-phase heat treatment [8]. According to the manufacturer, 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. Gambarini *et al.* [8] report that the post-grinding heat treatment seems not only to improve flexibility of NiTi rotary instruments, but also eliminate many drawbacks of the grinding process and provide a superior mechanical resistance as well. 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, K3 and K3XF [7, 9, 10]. Although K3XF showed improved cyclic fatigue resistance without degradation of torsional properties, the fundamental mechanism for this improved performance is unknown and the relationship between thermal behavior and mechanical properties of K3XF instruments has not been investigated nor clarified. Recently, Pérez-Higueras *et al.* [11] compared the cyclic fatigue resistance using two different motions (continuous rotation and reciprocation) among K3, K3XF and TF files. The manufacture of TF files includes three methods: R-phase heat treatment, twisting of the metal wire, and special surface conditioning. K3XF files were significantly more resistant than TF and K3. Under reciprocating motion, the cyclic fatigue resistance was greater for all files.

Another point that deserves attention is the sterilization step, where the instruments are usually held for circa 12 minutes at 134 °C. Several authors have reported that the transformation temperatures are not affected by the thermal cycles of sterilization [12, 13].

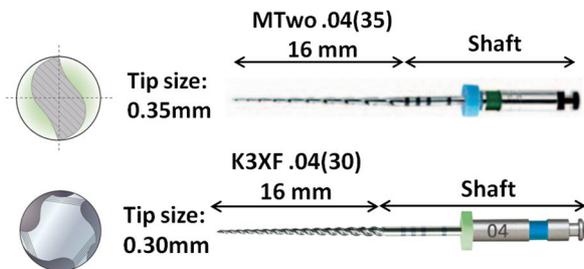
The purpose of this study was to evaluate the effect of heat treatment on the transformation temperatures of popular NiTi rotary files (K3 and MTwo files) and compare them with more recent K3XF files, using DSC (differential scanning calorimetry) and X-ray diffraction (XRD) to investigate the stress-induced martensite transformation on the files under flexion.

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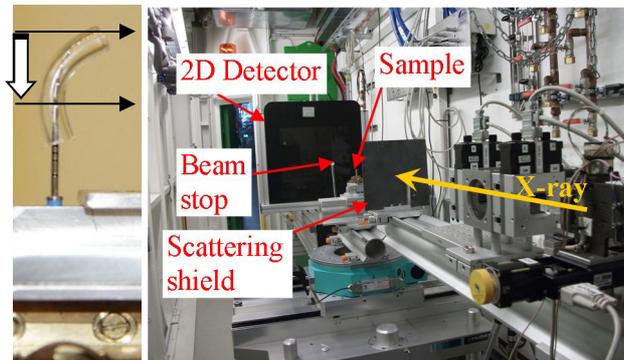
## 2 Materials and Methods

Three brands of rotary nickel-titanium endodontic instruments with some comparable geometrical features but different cross-section geometries (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). Fig. 1 shows the geometry of these files.

The instruments were analyzed under the following conditions: i) as-received (AR), ii) heat treated at 350°C, iii) heat treated at 400°C. The heat treatment (HT) was performed by holding the specimens at desired temperature for 1 hour and subsequently quenching them into water at room temperature (RT). The transformation temperatures were determined by DSC in which the shafts of the instruments were cut into three pieces weighing approximately 50 mg and placed in an aluminum crucible in the measuring chamber of a DSC (Netzsch DSC 204 F1 Phoenix) apparatus. With an empty aluminum 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 10 °K min<sup>-1</sup>. The temperature range of the DSC analysis was set between -150 and +100 °C. The  $A_{s_s}$ ,  $A_{f_s}$ ,  $M_s$ ,  $M_f$ ,  $R_s$ , and  $R_f$  (where applicable) are the transformation temperatures, where the subscripts s and f stand for start and finish of the austenite (A), martensite (M) and R-phase (R) transformations. These temperatures were determined by the intersection of an extrapolated baseline and the maximum gradient line of the DSC curve.

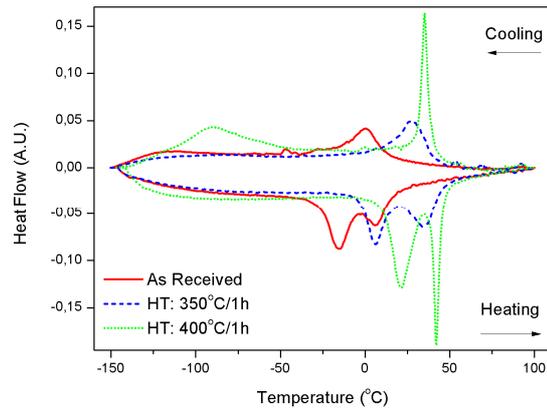


**Figure 1.** Overall geometry of the files studied (K3 has the same geometry as K3XF).

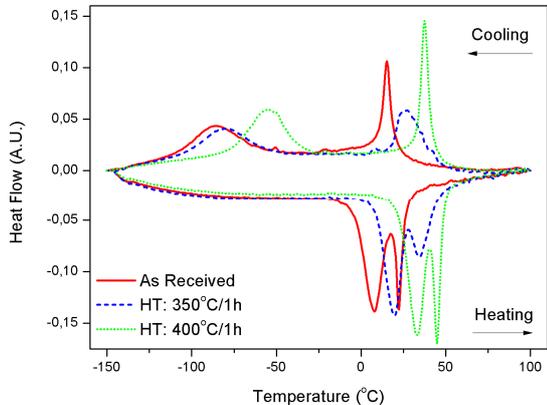


**Figure 1.** Mounting of the XRD measurements: (a) relative position of bent files and incident beam, (b) overall view, with 2D detector at the bottom.

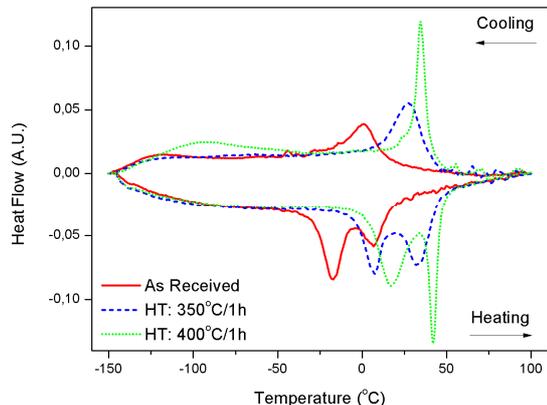
X-ray diffraction was performed on AR files comparing unbent files with bent files. The bent endodontic files were fixed by the handle, and a glass tube with a radius of curvature of 10 mm imposed a controlled curvature. XRD analysis was performed at beamline P07 of PETRA-III (DESY, Hamburg), using synchrotron radiation (87 keV/0.1422 Å) in transmission mode, and the incident beam was perpendicular to the bending axis of the file. Files were scanned from tip to handle using a beam spot of 50 x 50 μm<sup>2</sup> and with a spot spacing of 0.25 mm. A 2D-detector MAR345 placed at a distance of 1090 mm from the sample was used to image the Debye-Scherrer rings. These rings were then integrated from 0 to 360° using the software Fit2D. HT files were also compared with AR files in the unbent state.



**Figure 2.** DSC cooling and heating curves for K3 files.



**Figure 3.** DSC cooling and heating curves for K3XF files.



**Figure 4.** DSC cooling and heating curves for MTwo files.

### 3 Results and Discussion

#### 3.1 Differential Scanning Calorimetry measurements

DSC curves of the shaft region of K3, K3XF and MTwo AR and HT files are shown in Fig. 2, 3, and 4, respectively. A pair of endothermic peaks can be seen on the heating curve of the NiTi rotary files between -30 and 25 °C. These peaks correspond to the initial transformation of martensite to R-phase at lower temperatures, followed by transformation at higher temperatures of R-phase to austenite, which is completed at approximately 25°C for the AR files. The transformation temperatures for the three files tested are shown on Table 1. MTwo instrument had the highest  $A_f$  transformation temperatures.

The DSC results showed that all instruments in the HT condition have an  $A_f$  temperature above 37 °C, which is the assumed temperature of the oral environment. A mixture of R-phase and austenite shall then be during their clinical use at oral temperature, with the superelastic properties being dependent on the ration austenite/R-phase during clinical application.

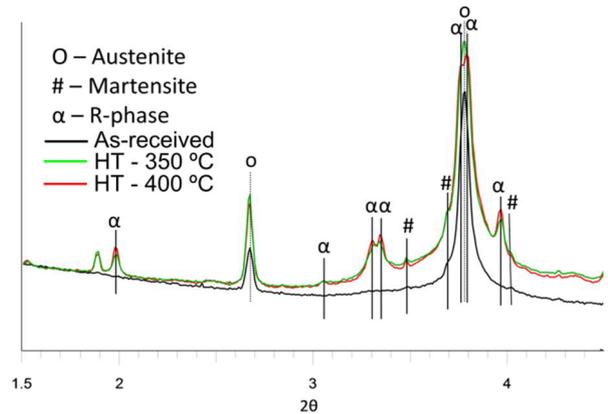
**Table 1.** Phase transformation temperatures, in °C

		Cooling				Heating				
		HT	R <sub>s</sub>	R <sub>f</sub>	M <sub>s</sub>	M <sub>f</sub>	R <sub>s</sub>	R <sub>f</sub>	A <sub>s</sub>	A <sub>f</sub>
MTwo	AR	18.1	-19.9	-80	< 150	-28.4	4.9	-12.5	25.6	
	350° C/1h	41.9	7.4	-100	< 150	-7.1	17.7	16.2	44.9	
	400° C/1h	43.9	20.9	-50	< 150	-0.6	38.6	36.8	47.9	
K3	AR	13.8	-29.7	-80	< 150	-32.2	-1.4	-14.3	17.8	
	350° C/1h	41.8	0.8	-110	< 150	-4.7	19.6	16.8	45.8	
	400° C/1h	43.1	23.6	-60	< 150	7.8	37.7	39.1	47.1	
K3XF	AR	24.8	1.8	-59.7	-115.6	-10.7	19.1	18.3	28.3	
	350° C/1h	43.6	16.1	-55.9	-111.3	4.1	29.2	22.3	46.6	
	400° C/1h	46.3	26.8	-30.6	-85.2	13.8	42.4	40.7	50.8	

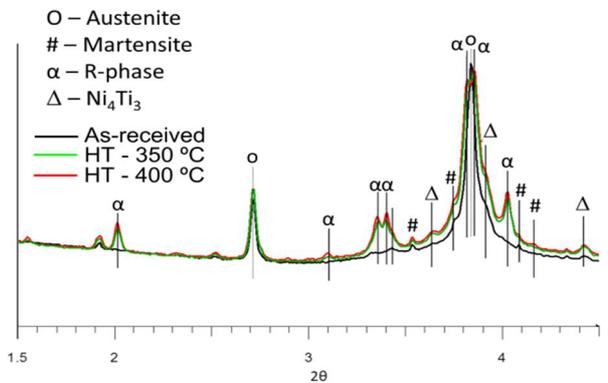
The  $A_f$  temperature for the AR K3 file was below RT while for AR K3XF and AR MTwo it was slightly above it. It has been reported that appropriate heat treatments can increase the transformation temperatures and improve the flexibility of endodontic files [14] and it has also been suggested that the heat treatments cause a release of crystal lattice defects and contributes to changes in the phase transformation temperatures [14, 15]. Compared with 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 350 °C in order to obtain transformation temperatures closer to RT. There can also be observed two endothermic peaks, indicating that the reverse transformation passes through the intermediate R-phase in all HT files.

#### 3.2 X-ray diffraction measurements

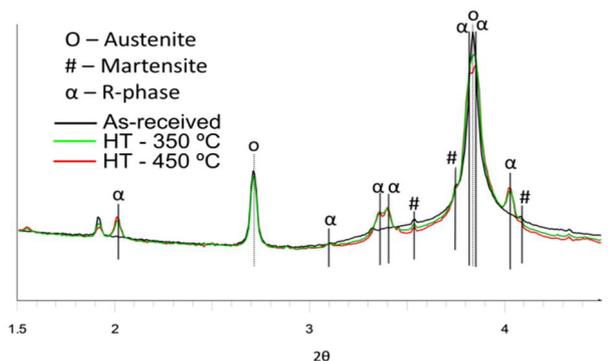
Compared to AR files, the XRD patterns of heat treated files show more R-phase and more intense martensitic peaks (Figs 5-7). The R-phase peaks are more intense in K3XF files when increasing the heat treatment temperature form 350 to 450 °C, suggesting an increase in flexibility. These results are consistent with the DSC analysis: (i) higher transformation temperatures for the HT files and (ii) two-step reverse transformation passing through the intermediate R-phase. R-phase and martensite are already present, at room temperature, in these files after heat treatment process.



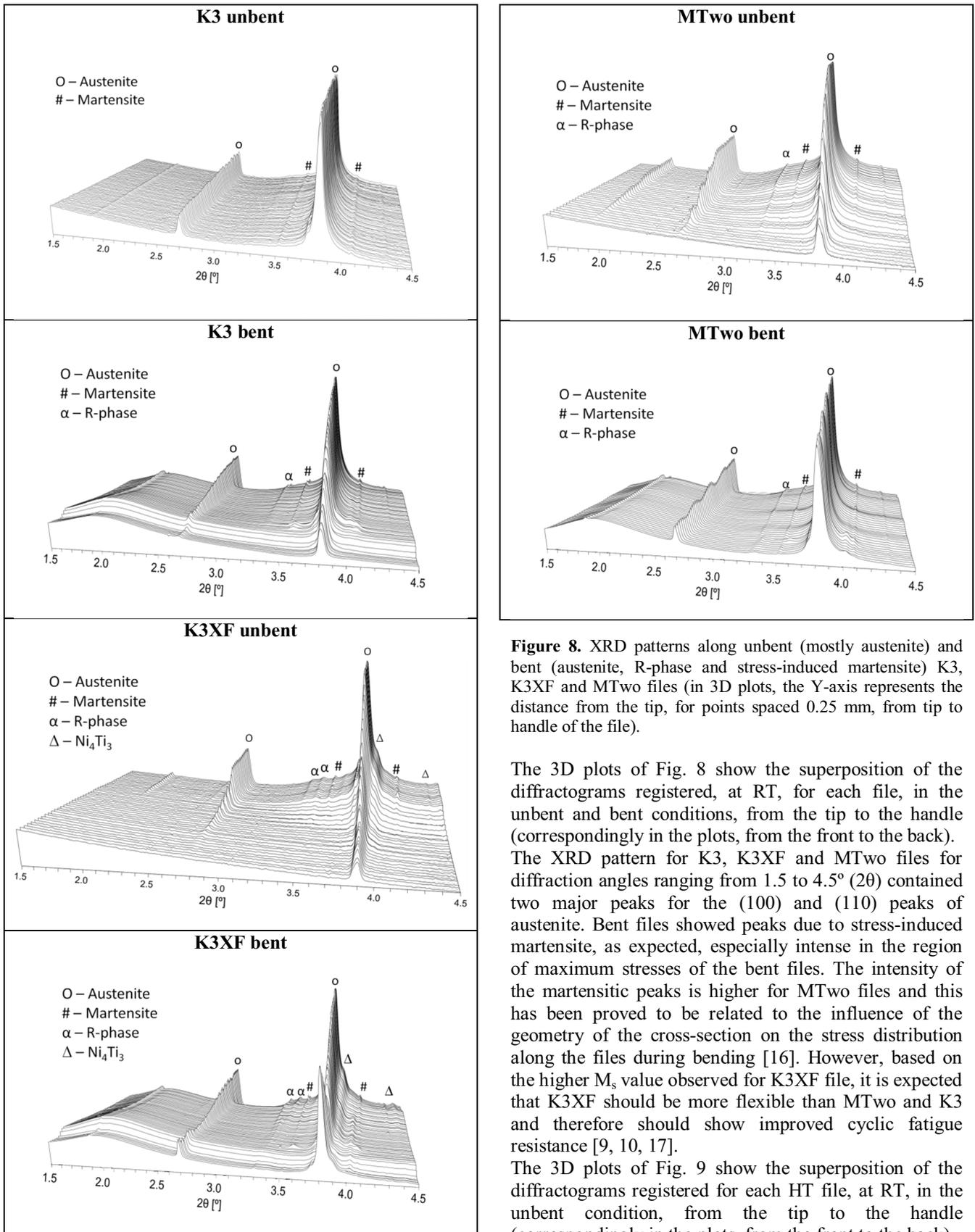
**Figure 5.** XRD scans of K3 files: AR (---); HT – 350°C (---), HT – 400°C (---) files.



**Figure 6.** XRD scans of K3XF files: AR (---); HT – 350°C (---), HT – 400°C (---) files.



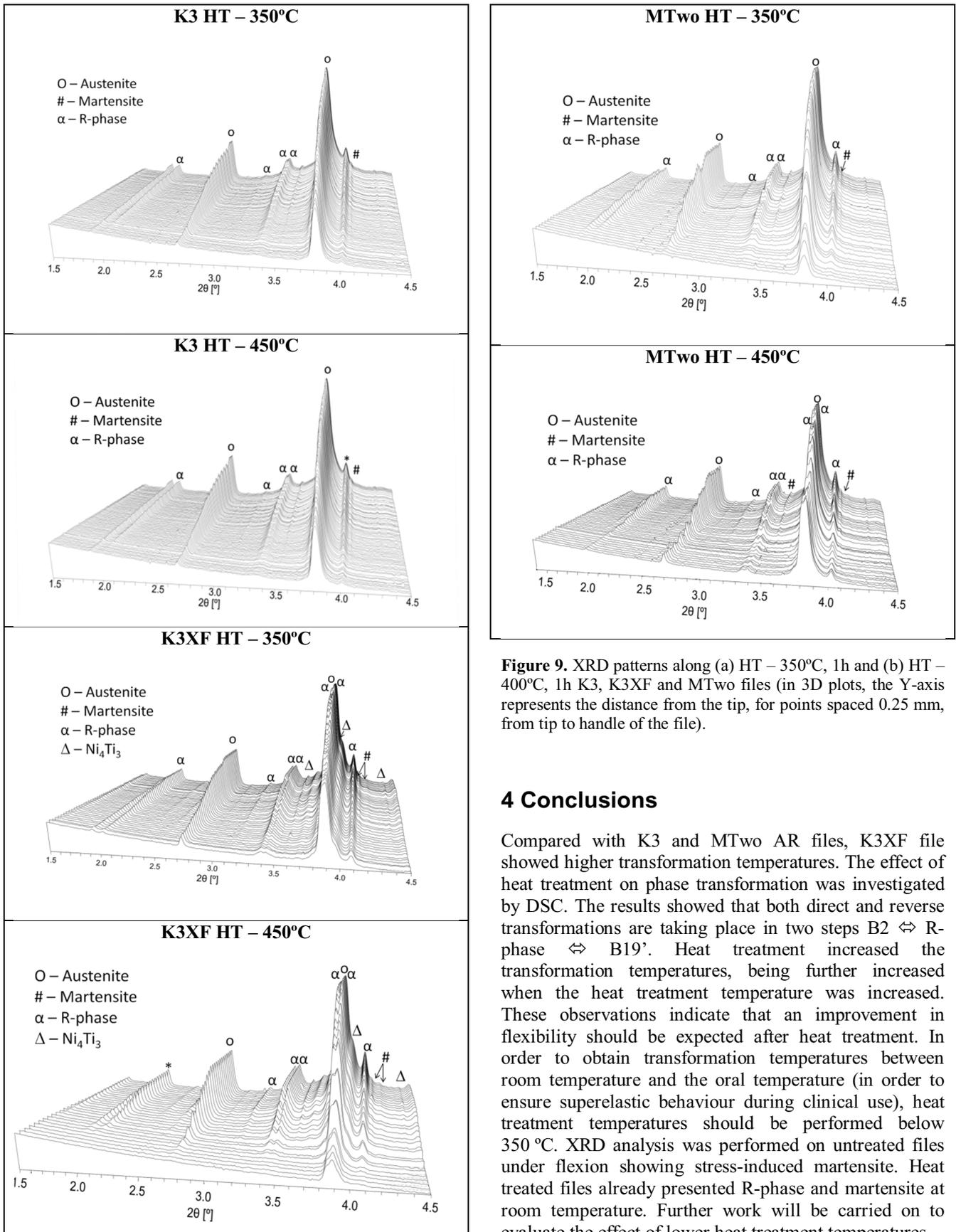
**Figure 7.** XRD scans of MTwo files: AR (---); HT – 350°C (---), HT – 400°C (---) files.



**Figure 8.** XRD patterns along unbent (mostly austenite) and bent (austenite, R-phase and stress-induced martensite) K3, K3XF and MTwo files (in 3D plots, the Y-axis represents the distance from the tip, for points spaced 0.25 mm, from tip to handle of the file).

The 3D plots of Fig. 8 show the superposition of the diffractograms registered, at RT, for each file, in the unbent and bent conditions, from the tip to the handle (correspondingly in the plots, from the front to the back). The XRD pattern for K3, K3XF and MTwo files for diffraction angles ranging from 1.5 to 4.5° ( $2\theta$ ) contained two major peaks for the (100) and (110) peaks of austenite. Bent files showed peaks due to stress-induced martensite, as expected, especially intense in the region of maximum stresses of the bent files. The intensity of the martensitic peaks is higher for MTwo files and this has been proved to be related to the influence of the geometry of the cross-section on the stress distribution along the files during bending [16]. However, based on the higher  $M_s$  value observed for K3XF file, it is expected that K3XF should be more flexible than MTwo and K3 and therefore should show improved cyclic fatigue resistance [9, 10, 17].

The 3D plots of Fig. 9 show the superposition of the diffractograms registered for each HT file, at RT, in the unbent condition, from the tip to the handle (correspondingly in the plots, from the front to the back).



**Figure 9.** XRD patterns along (a) HT – 350°C, 1h and (b) HT – 400°C, 1h K3, K3XF and MTwo files (in 3D plots, the Y-axis represents the distance from the tip, for points spaced 0.25 mm, from tip to handle of the file).

#### 4 Conclusions

Compared with K3 and MTwo AR files, K3XF file showed higher transformation temperatures. The effect of heat treatment on phase transformation was investigated by DSC. The results showed that both direct and reverse transformations are taking place in two steps  $B2 \leftrightarrow R\text{-phase} \leftrightarrow B19'$ . Heat treatment increased the transformation temperatures, being further increased when the heat treatment temperature was increased. These observations indicate that an improvement in flexibility should be expected after heat treatment. In order to obtain transformation temperatures between room temperature and the oral temperature (in order to ensure superelastic behaviour during clinical use), heat treatment temperatures should be performed below 350 °C. XRD analysis was performed on untreated files under flexion showing stress-induced martensite. Heat treated files already presented R-phase and martensite at room temperature. Further work will be carried on to evaluate the effect of lower heat treatment temperatures.

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