

## Effect of Temperature on Cyclic Fatigue Resistance of R-phase Technology and Conventionally Produced Nickel-titanium Alloys

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### Abstract

This study evaluated the effect of temperature on the cyclic fatigue resistance of nickel-titanium rotary files produced by conventional and R-phase technology and to determine the austenite finishing temperature of these nickel-titanium alloys. The cyclic fatigue resistance of K3 and K3XF files was determined at room and body temperatures using a model in which the canal shape was similar to that of the file. The austenite finishing temperatures of the K3 and K3XF files were also investigated. The cyclic fatigue resistance of the conventional or R-phase technology alloys was significantly reduced at body temperature. The cyclic fatigue resistance of the R-phase technology alloy was approximately 2-fold higher compared with the conventional alloy in both temperatures. The austenite finishing temperature of each alloy was lower than body temperature. A cooling strategy may increase the files' cyclic fatigue resistance which benefits shaping challenging curve canals.

**Keywords :** Cyclic fatigue Resistance, Differential scanning calorimetry, Nickel-titanium rotary, R-phase, temperature

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### Introduction

The goal of endodontic treatment is to prevent and intercept periapical pathosis. Root canal treatment aims to reduce the intraradicular microbes as much as possible and seal the root canal system to establish an appropriate environment for periapical healing. Mechanical instrumentation significantly eradicates intraradicular microbes<sup>1,2</sup> and enhances antimicrobial irrigation flow.<sup>2</sup> Endodontic instruments have been developed to facilitate root canal preparation.

Nickel-titanium (Ni-Ti) alloys were first used as root canal files in 1980.<sup>3</sup> These files are widely used due to their superior properties compared with stainless steel files, e.g. flexibility to maintain the original curve of the root canal<sup>4,6</sup>, less errors and preparation time.<sup>5</sup> Ni-Ti alloy has three microstructure phases: austenite, martensite, and R-phase.

Austenite, which has a B2 cubic crystal structure, is quite strong and hard. In contrast, martensite, which has a monoclinic B'19 structure, is soft, ductile, and flexible.

R-phase is the intermediate phase between the austenite and the martensite phases, and has a rhombohedral structure. A Ni-Ti rotary file is more flexible when it is in its martensitic microstructure compared with the austenitic microstructure.<sup>7</sup> Furthermore, the martensitic microstructure in Ni-Ti alloys results in high cyclic fatigue resistance.<sup>8</sup>

Manufacturers have developed Ni-Ti rotary files to be in martensitic microstructure in clinical situations using heat-treatment procedures. Heat treatment is the process of heating the alloy to a specific recovery temperature to improve the alloy's physical properties and increase the transformation temperature, thus, increasing flexibility.<sup>9,10</sup> Heat treatment can be done pre- or post-machining.

The austenitic and martensitic microstructure ratio is temperature-dependent. When the temperature rises, more austenitic and less martensitic microstructure is present and vice versa.<sup>11</sup> Complete austenitic microstructure is present when the alloy is heated beyond a specific temperature known as its austenite finishing temperature (Af temperature).<sup>11</sup> Previous studies demonstrated that increased temperature tended to decrease cyclic fatigue resistance, including conventional Ni-Ti or heat treated Ni-Ti alloys, such as R-phase, CM wire, and blue and gold wire.<sup>12-17</sup> To shape a challenging curved canal, high cyclic fatigue resistance of the Ni-Ti rotary file is mandatory for successful root canal preparation without file separation.

K3XF (SybronEndo) is made using R-phase-technology. The Ni-Ti file receives a proprietary thermal treatment after grinding. This treatment modifies the crystalline defects that occur during grinding and increases file flexibility.<sup>11,18</sup> Previous studies demonstrated that thermal-treated R-phase Ni-Ti files (K4 or K3XF) had a higher cyclic fatigue resistance compared with its prototype (K3), which is produced using conventional Ni-Ti alloy.<sup>18,19</sup> However, these studies were performed at room temperature. Later studies investigated the cyclic fatigue resistance of K3XF and K3 files at different temperatures. However, the cyclic fatigue testing model did not resemble the file dimension.<sup>16,17</sup> Currently, there are no reports on the cyclic fatigue resistance of R-phase using different temperatures and an appropriate

model. The aim of this study was to evaluate the effect of temperature on the cyclic fatigue resistance of Ni-Ti rotary files produced from conventional or R-phase technology alloys at simulated room and body temperature using an accurate simulated curved canal and determine the Af temperature of these Ni-Ti alloys. The null hypothesis was that the cyclic fatigue resistance at room and body temperature is not different in either the conventional or R-phase technology produced alloys.

## Materials and Methods

### Cyclic fatigue resistance testing

The sample size for each group was calculated using the equation for testing three independent means as previously described.<sup>17</sup> The type 1 error and type 2 error was 0.05 and 0.2, respectively. This calculation indicated that the minimum sample size for each group was six. We added 66 % more samples to compensate for sampling error and increase the level of precision. Thus, the number of samples for quantitative data in each group was ten. Twenty #50 (0.06 taper) K3XF [SybronEndo, Orange, CA] and K3 [SybronEndo, Orange, CA] files were evaluated using a dental operating microscope [OPMI PROERGO, Zeiss, Germany] to confirm that the files had no visible defects. Files with a defect were discarded and replaced with a new file.

The cyclic fatigue resistance of each file was tested using a steel artificial curved canal model with a 60° curvature angle and 5 mm radius of curvature. The shape and taper of the artificial canal that resembled the file dimension was produced by a 3D printing machine [CNC Milling sister SD543, MNP, Malaysia]. The rotary file was scanned and a three-dimensional blueprint was created. The steel block was then precisely milled based on the blueprint. The artificial canal depth was one third deeper than the file diameter for its entire length to prevent the file from dislodging and faced with tempered glass for file stability while rotating in the canal and for directly visualizing file fracture.

Ten files of each type were randomized into two groups that were tested at room temperature (20°±1°C)

or simulated body temperature ( $37^{\circ}\text{C}\pm 1^{\circ}\text{C}$ ). The model was set in a water-controlled temperature glass container. Two thermometers were used to confirm the accuracy of the experimental temperatures; one was set at the top of the container, while the other was set at the bottom. The files were activated in the cyclic fatigue testing model using a torque-control motor [VDW Silver Reciproc motor, VDW] at 300 rpm and were recorded using a dental operating microscope video camera until each file fractured. The number of cycles to failure (NCF) was calculated by multiplying the time to failure in seconds by five (cycles per second). The mean and standard deviation of the NCF in each group was also calculated. The data was tested for a normal distribution using the Kolmogorov-Smirnov test. The differences between within and between alloy groups were assessed with the Mann-Whitney U test. Significance was set at a 95% confidence level.

#### Differential scanning calorimetry (DSC) analysis

Two new K3 and K3XF files were cut into 2 mm samples using a slow-speed diamond saw with water coolant [Isomet 1000; Buehler, Lake Bluff, IL, USA]. The

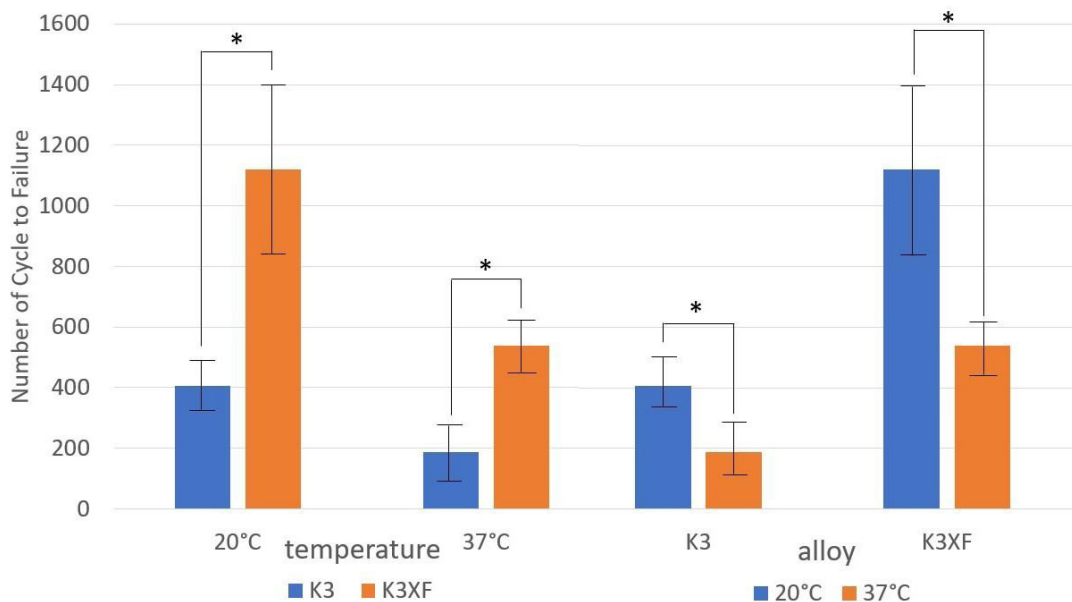
samples were placed in a differential scanning calorimeter [model DSC822<sup>o</sup>, METTLE TOLEDO, Langacher, Switzerland]. Nitrogen purge gas at a flow rate of 50 mL/min was used. The samples were heated from ambient temperature to  $100^{\circ}\text{C}$  then cooled to  $-150^{\circ}\text{C}$  at a rate of  $-10^{\circ}\text{C}/\text{minute}$  followed immediately by a heating cycle at  $10^{\circ}\text{C}/\text{minute}$  up to  $100^{\circ}\text{C}$ . The heating and cooling cycles were repeated three times. The data were automatically plotted as a DSC graph. The Af temperature was measured by drawing the intersection of a tangent line to the right side of the endothermic peak for total heat flow with the adjacent base line<sup>10</sup>.

## Results

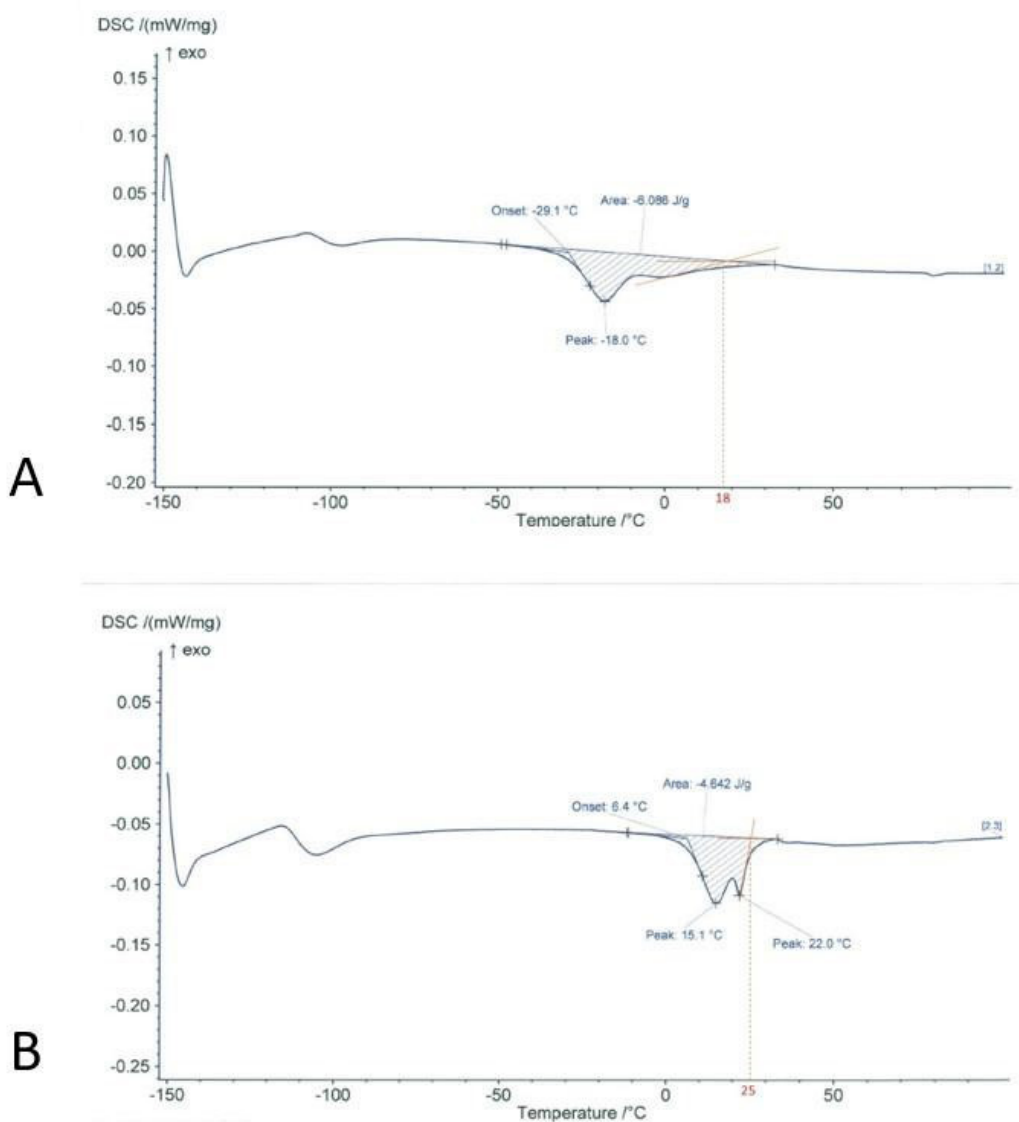
The mean K3XF and K3 files' NCF and standard deviations (SD) were determined (Fig. 1). The mean K3XF file NCF was significantly higher compared with the K3 file NCF at both temperatures. Within the same alloy type, the mean NCF at body temperature was significantly lower than at room temperature.

#### Transformation temperature

The DSC plots for both files are presented in (Fig. 2).



**Figure 1** The number of cycles to failure (NCF) of different NiTi rotary files at different temperatures. The sign \* indicate statistically differences between different groups



**Figure 2** (A.) Differential scanning calorimetry plot of K3 (B.) Differential scanning calorimetry plot of K3XF

The K3 files exhibited a classic phase transformation plot with a single endothermic peak (Fig. 2A). Conversely, the K3XF files exhibited two endothermic peaks; the first peak represented the transformation from the martensitic phase to the R-phase and the second peak represented the transformation from R-phase to the austenitic phase (Fig. 2B). The  $A_f$  temperature of the K3 and K3XF files was 18°C and 25°C, respectively.

## Discussion

This study investigated the effect of temperature on the cyclic failure resistance and  $A_f$  temperature of two

different Ni-Ti alloys. The results indicated that the fatigue resistance at body temperature was approximately 50 % lower than that at room temperature for both alloys. Based on these findings, the null hypothesis was rejected. It was also found that the  $A_f$  temperature of the K3XF files was higher compared with the K3 files.

The reduced K3XF file's cyclic fatigue resistance at body temperature resulted from its  $A_f$  temperature, which was between room and body temperatures. Thus, these files were mainly in the austenite phase at body temperature. This phase made the files less flexible and easier to fracture. Our results agreed with a previous study<sup>17</sup>

that exhibited a significant difference in NCF between temperatures. In contrast, Shen *et al.*<sup>16</sup> found similar cyclic fatigue reduction results, however the difference was not significant. Both of these previous studies tested different types of files using only one artificial canal model. Therefore, the disparate results may be due to the differences between the simulated models.

The K3 files had an Af temperature that was lower than room and body temperatures. However, the cyclic fatigue resistance of these files was lower at body temperature. These results agreed with other conventionally produced Ni-Ti alloys from different manufacturers<sup>13</sup>. Interestingly, a previous study using micro-x-ray diffraction and scanning electron microscopy demonstrated that some martensitic microstructure was found when Ni-Ti files were above its Af temperature.<sup>20</sup> These results may explain our findings. This stable martensitic microstructure did not undergo transformation and might gradually turn to austenitic microstructure when heated from room temperature to body temperature.

Previous studies found that K3XF files had a higher cyclic fatigue resistance compared with K3 files at room temperature.<sup>18,19</sup> These findings can be explained by the reduced lattice defects and microstructure internal stress resulting from thermal treatment. Therefore, generating plastic deformation is more difficult.<sup>9,21</sup> Thus, thermal treatment of an alloy produces a high cyclic fatigue resistance file.

Many studies demonstrated that file design, such as cross-section, shape, and taper, influenced cyclic fatigue resistance.<sup>22-24</sup> Thus, when comparing two file systems with different geometric designs, the results are difficult to interpret and does not allow for identifying the true factors that influenced the results or whether they were caused by taper, size, or alloy differences. In this study, K3 files were compared with K3XF files because they share the same geometry, cross-section, taper, and diameter. Every factor was controlled. The only different factor was the alloy used in the manufacturing process. Thus, we can conclude that the different cyclic fatigue resistances resulted from either the different alloys or temperatures.

In previous studies, the cyclic fatigue testing models varied, such as a curved metal tube<sup>25</sup>, rotating between concave and convex-tempered steel rods<sup>26</sup>, rotating against a sloped plane<sup>27</sup>, and rotating between two cylinder pins.<sup>28</sup> The disadvantage of these models was the uncontrolled trajectory of the file because the model was not tapered similarly to the tested file.<sup>29</sup> Thus, the best available model was a metal artificial canal that corresponded with the file's shape, taper, and size that constrained the file in a precise trajectory. Using only one model to test different files resulted in imprecise file trajectories. Some files likely loosely fit the canal, while others fit more accurately. The radius of curvature of different files will be under or overstated based on the file trajectories. Moreover, a file that fits loosely inside the model may exhibit a greater or lesser degree of curvature depending on its flexibility. These differences explain why we cannot compare the results of the same file with the same degree and radius of curvature tested using different model designs between studies. The present study used a steel artificial canal that was precisely constructed with 3D printing technology. Therefore, it was the most reliable cyclic fatigue testing model available for comparative assessment. Specific model designs are required when testing files with different designs. Thus, comparing the different file systems requires a precise model for each file.

Previous studies did not evaluate the effect of environmental temperature on the cyclic fatigue resistance of K3 and K3XF files.<sup>18,19</sup> Although two studies evaluated the files at body temperature, these studies used only one model that did not closely resemble the files' dimensions and tested files with different geometries.<sup>16,17</sup> Thus, cyclic fatigue resistance studies using an artificial canal fit to the file's shape conducted at body temperature might be a better reference for clinical application.

To date, there is no conclusion on real clinical root canal temperature. Hemptinne *et al.*<sup>30</sup> investigated irrigating solution temperature in a root canal at every second while irrigating with room temperature sodium hypochlorite (NaOCl) solution (19.4°C) and preheated NaOCl solution (66°C). The result showed that irrigating

solution temperature in the root canal always turned to approximately 35°C after increasing or decreasing the temperature within a period of time. It could be assumed that root canal temperature is approximately the same and close to body temperature. In a clinical situation, while the rotary file works in a root canal, it cuts dentin and has friction against the canal wall. The temperature of the instrument definitely increased more or less.

Single-use of file was recommended by most of the manufacturers. However, reusing files is common in developing countries because of economic factors. Understanding the true potential of alloy will help clinicians to choose the instrument with high performance which can reduce error in their practice. Furthermore, Arens *et al.*<sup>31</sup> found 0.9 % fracture incidence in new files that were used for the first time. Thus, choosing instruments that are produced by novel alloy may benefit in safety not only for reusing files but also reduce risk in new files that are intended to shape the extremely challenged canal which generates intense cyclic fatigue to the instrument.

Previous studies<sup>12-17</sup>, including the present study, found that body temperature decreased the cyclic fatigue resistance of the files. We recommend that the clinician choose the higher Af temperature NiTi rotary file especially the one in which its Af is higher than body temperature. This will minimize the chance of fracture while using it in a root canal from its martensitic properties. Cooling the environment temperature increased the cyclic fatigue resistance of most of the Ni-Ti alloys. Future studies should evaluate using a cooling strategy, which might increase the files' cyclic fatigue resistance in challenging shaped canals.

## Conclusion

The cyclic fatigue resistance of the conventional or R-phase technology alloys was significantly reduced at body temperature. The cyclic fatigue resistance of the R-phase technology alloy was approximately two-fold higher compared with the conventional alloy in both temperatures. The austenite finishing temperature of each alloy was lower than body temperature.

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