The influence of heat treatment on the mechanical properties of Ni-Ti file materials

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The purpose of this study was to investigate the influence of heat treatment on the mechanical properties of Ni-Ti file materials. Ni-Ti wire (1.00 mm ø) was processed into a conical shape with 0.30-mm diameter tip and 0.06 taper. Specimens were heated for 30 min at 300, 400, 450, 500 or 600°C. Non-heated specimens were used as controls. DSC, a cantilever-bending test and cyclic fatigue test were performed. Mₛ and Aᶠ for groups 400 and 450 were higher than those for others (p<0.05). The load/deflection ratios of groups 400, 450 and 500 were lower than that of group 600 (p<0.05). The bending load values at 2.0-mm deflection of groups 400, 450 and 500 were lower than those of group 300 and the control group (p<0.05). The NCFs of groups 400, 450 and 500 exceeded that of group 600(p<0.05). Changes in flexibility with heat treatment could improve the cyclic fatigue properties of Ni-Ti instruments.

Keywords: Bending property, Cyclic fatigue property, Heat treatment, Ni-Ti alloy, Transformation behavior

INTRODUCTION

Ni-Ti instruments were introduced to develop root canal shaping1). Ni-Ti endodontic instruments exhibit greater flexibility compared with stainless-steel instruments. The flexibility of Ni-Ti alloy offers clinical advantages in the shaping of curved canals2,3).

Ni-Ti alloy possesses unique mechanical properties. These include shape-memory effect and super-elasticity4-6). These special characteristics are derived from martensitic and austenitic transformations, with super-elasticity being observed at temperatures above the reverse transformation finishing point, Aᶠ7).

Phase transformation behavior has an impact on the mechanical properties of Ni-Ti instruments7-11) and is easily influenced by factors including chemical composition, heat treatment and manufacturing processes4,6). Previous studies reported heat-treated Ni-Ti alloy instruments had greater flexibility than instruments that had not been heat treated7,11,12). Recently, heat treatment of Ni-Ti alloys (e.g., M-Wire: Dentsply Tulsa Dental Specialties, Tulsa, OK13), and R-phase wire: SybronEndo, Orange, CA14) has been used, as part of instrument manufacture, to enhance the mechanical properties.

However, Ni-Ti endodontic instruments have a high risk of fracture in use because of cyclic fatigue15) as a result of the continuous rotation of the instrument in a curved canal. As Ni-Ti instruments may show no visible signs of permanent deformation during cyclic fatigue, instrument separation may occur unexpectedly16). Many studies reported the cyclic fatigue properties of products on the market17,18). However, there is little information available on the effect of heat treatment on the cyclic fatigue properties of Ni-Ti endodontic instruments19). The aim of this study was to investigate the influence of heat treatment on the cyclic fatigue properties of Ni-Ti endodontic instruments fabricated from Ni-Ti alloy wires. The effects of heat treatment on transformation behavior and bending properties were also examined and related to the fatigue properties.

MATERIALS AND METHODS

Specimens
Ni-Ti alloy wire (1.00 mm diameter, Ti-50.95 at% Ni, NT-N, Furukawa Techno Material, Kanagawa, Japan) with super-elasticity and memorized straight shape was used in this study. Conical specimens with a 0.30-mm tip diameter and 0.06 taper, as shown in Fig. 1, were ground and not surface-treated.

Heat treatment conditions
A total of 144 specimens were randomly divided into six groups for heat treatment, and encapsulated in silica glass tubes in a vacuum of less than 10⁻³ Pa. They were heated for 30 min at 300°C (group 300), 400°C (group 400), 450°C (group 450), 500°C (group 500) and 600°C (group 600), followed by quenching in iced water with simultaneous fracture of the glass tubes. Specimens without heat treatment were served as controls.

Differential scanning calorimetry (DSC)
Seven specimens from each group were subjected to this...
test. Phase transformation behavior of the specimens was measured by DSC. The conical sections of the specimens weighing approximately 20 mg were used. The specimens were cut into 2- to 3-mm-long pieces and sealed in aluminum cells. Then they were placed in the measuring chamber of a differential scanning calorimeter (DSC-60; Shimadzu, Kyoto, Japan), and the chamber was filled with argon gas. An empty aluminum cell was used as a reference. Liquid nitrogen acted as the coolant, and the heating and cooling rates were set to 0.33°C/s. The temperature was increased from room temperature to 100°C, and then decreased to −100°C to obtain the cooling curve. The temperature was then increased back to 100°C and the heating curve recorded. The interpretation of the DSC curves was based on previous studies\(^1^1\), in which the transformation temperatures were obtained from the intersections between extrapolations of the baseline and the maximum-gradient line of the lambda-type DSC curve. The martensitic transformation starting (\(M_s\)) and finishing (\(M_f\)) points and the reverse transformation starting (\(A_s\)) and finishing (\(A_f\)) points were determined.

**Bending tests**

Seven specimens from each group were examined using a cantilever-bending test device. The detail of the equipment is described elsewhere\(^9\),\(^1^1\),\(^1^2\). The specimen was mounted on a movable stage, and the temperature of the specimen and equipment was kept at 37°C. The specimen was loaded (1.0 mm/min) until the deflection reached 3.0 mm and then unloaded. The distance between the clamp edge and the file tip was 9.5 mm, and the initial loading point was 3.0 mm distant from the specimen tip. From load-deflection curves, the load-deflection (L/D) ratio of the initial linear part was calculated. The bending load was measured at 2.0-mm deflection, which corresponded to the super-elastic range.

**Cyclic fatigue tests**

Cyclic fatigue testing was performed using a stainless steel three-pin device. The experimental settings were based on previous studies\(^2^0\). The specimen was set and protruded from the lowest pin by 2 mm, and then deflected horizontally by either 1.0 mm (R10, 10 mm radius of curvature, 15° angle of curvature) or 1.5 mm (R5, 5 mm radius of curvature, 20° angle of curvature). Ten specimens of each group were tested for each condition, and rotated at 300 rpm with a Dentaport (J Morita, Kyoto, Japan). The time until fracture was recorded and the number of cycles to fracture (NCF) was then calculated. All measurements were performed at 37°C. Silicone oil (KF-96-100CS; Shin-Etsu Chemical Co., Tokyo, Japan) was used as a lubricant to minimize friction and heat generation.

**Data analysis**

For evaluation of the transformation temperatures, bending load values and NCF, ANOVA was used for detection of differences amongst the groups. Differences between the groups were analyzed by Tukey-Kramer’s post hoc test, and the statistical significance was set at \(p=0.05\).

**RESULTS**

**DSC**

Figure 2 shows typical DSC curves obtained from each group. The upper and lower curves in each trace are cooling and heating runs, respectively. Exothermic transitions are upwards.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Heat Flow (mW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-80</td>
<td>0</td>
</tr>
<tr>
<td>-40</td>
<td>0</td>
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<tr>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>40</td>
<td>0</td>
</tr>
<tr>
<td>50</td>
<td>0</td>
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<tr>
<td>60</td>
<td>0</td>
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<tr>
<td>70</td>
<td>0</td>
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<tr>
<td>80</td>
<td>0</td>
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<td>90</td>
<td>0</td>
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<td>100</td>
<td>0</td>
</tr>
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</table>

Fig. 2 Typical DSC curves for each group. The upper and lower curves in each trace are cooling and heating runs, respectively. Exothermic transitions are upwards.
Table 1  Phase transformation temperatures for each group (mean±standard deviation, n=7)

<table>
<thead>
<tr>
<th></th>
<th>Ms (ºC)</th>
<th>Mf (ºC)</th>
<th>As (ºC)</th>
<th>Af (ºC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>20.0±4.4&lt;sup&gt;a&lt;/sup&gt;</td>
<td>-18.7±2.3&lt;sup&gt;a&lt;/sup&gt;</td>
<td>-19.3±2.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>34.8±2.7&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>400</td>
<td>39.4±2.3&lt;sup&gt;b&lt;/sup&gt;</td>
<td>8.5±2.4&lt;sup&gt;b&lt;/sup&gt;</td>
<td>-0.4±1.6&lt;sup&gt;b&lt;/sup&gt;</td>
<td>52.6±1.1&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>450</td>
<td>35.3±2.1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>12.2±2.6&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.0±1.3&lt;sup&gt;b&lt;/sup&gt;</td>
<td>47.8±3.2&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>500</td>
<td>24.5±1.8&lt;sup&gt;b&lt;/sup&gt;</td>
<td>8.3±1.2&lt;sup&gt;b&lt;/sup&gt;</td>
<td>-1.9±1.2&lt;sup&gt;b&lt;/sup&gt;</td>
<td>38.7±3.5&lt;sup&gt;b&lt;/sup&gt;</td>
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<td>600</td>
<td>-8.5±9.9&lt;sup&gt;c&lt;/sup&gt;</td>
<td>-67.0±6.7&lt;sup&gt;c&lt;/sup&gt;</td>
<td>-22.7±4.2&lt;sup&gt;c&lt;/sup&gt;</td>
<td>30.2±4.3&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td>control</td>
<td>20.9±11.4&lt;sup&gt;a&lt;/sup&gt;</td>
<td>-26.3±4.7&lt;sup&gt;d&lt;/sup&gt;</td>
<td>-23.2±2.7&lt;sup&gt;d&lt;/sup&gt;</td>
<td>28.7±4.2&lt;sup&gt;d&lt;/sup&gt;</td>
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Different superscript letters in each column represent statistically significant results.

Table 2  L/D ratios and bending load values at a deflection of 2.0 mm for each group (mean±standard deviation, n=7)

<table>
<thead>
<tr>
<th></th>
<th>L/D ratio (N/mm)</th>
<th>2.0-mm deflection (N)</th>
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<tbody>
<tr>
<td>300</td>
<td>0.69±0.15&lt;sup&gt;a&lt;/sup&gt;</td>
<td>9.58±1.29&lt;sup&gt;c&lt;/sup&gt;</td>
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<tr>
<td>400</td>
<td>0.47±0.06&lt;sup&gt;b&lt;/sup&gt;</td>
<td>7.55±0.64&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>450</td>
<td>0.50±0.08&lt;sup&gt;b&lt;/sup&gt;</td>
<td>7.45±0.69&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>500</td>
<td>0.60±0.07&lt;sup&gt;b&lt;/sup&gt;</td>
<td>7.79±0.64&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>600</td>
<td>0.85±0.12&lt;sup&gt;c&lt;/sup&gt;</td>
<td>8.68±1.30&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>control</td>
<td>0.75±0.09&lt;sup&gt;c&lt;/sup&gt;</td>
<td>10.67±0.81&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Different superscript letters in each column represent statistically significant results.

Table 3  Number of cycles to failure for each group, R10 and R5 (mean±standard deviation, n=10)

<table>
<thead>
<tr>
<th></th>
<th>R10</th>
<th>R5</th>
</tr>
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<tbody>
<tr>
<td>300</td>
<td>757.0±157.8&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>161.2±29.8&lt;sup&gt;ac&lt;/sup&gt;</td>
</tr>
<tr>
<td>400</td>
<td>871.2±127.2&lt;sup&gt;ac&lt;/sup&gt;</td>
<td>180.9±31.4&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>450</td>
<td>889.3±256.4&lt;sup&gt;ac&lt;/sup&gt;</td>
<td>189.1±22.4&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>500</td>
<td>966.6±271.3&lt;sup&gt;c&lt;/sup&gt;</td>
<td>176.8±35.1&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>600</td>
<td>588.5±133.5&lt;sup&gt;a&lt;/sup&gt;</td>
<td>128.3±20.8&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>control</td>
<td>637.2±165.4&lt;sup&gt;c&lt;/sup&gt;</td>
<td>101.8±34.9&lt;sup&gt;b&lt;/sup&gt;</td>
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</tbody>
</table>

Different superscript letters in each column represent statistically significant results.

Fig. 3  Typical load-deflection curves for each group.

**Bending tests**

Typical load-deflection curves for all groups, indicating super-elastic behavior, are shown in Fig. 3. The curves for all groups were initially linear, signifying elastic deformation. Above this range, the load level becomes almost constant, because of the stress-induced martensitic transformation.

During the unloading process, the load decreased rapidly and became constant, because of the reverse transformation. Table 2 presents the L/D ratios of all groups and the bending loads at 2.0-mm deflection. The L/D ratios of groups 400 and 450 were significantly lower than those of group 600 and the control group (p<0.05). The L/D ratio of group 500 was significantly lower than that of group 600 (p<0.05). The bending load values at 2.0-mm deflection of groups 400, 450 and 500 were significantly lower than those of group 300 and the control group (p<0.05).

**Cyclic fatigue tests**

The mean NCF values under both conditions are presented in Table 3. Under R10 condition, the NCF of group 500 was significantly greater than those of group 600 and the control group (p<0.05). The NCFs of groups 400 and 450 were significantly greater than that of group 600 (p<0.05). Under R5 condition, the NCFs of groups 400, 450 and 500 were significantly greater than those of group 600 and the control group (p<0.05).

**DISCUSSION**

**Transformation behavior**

The transformation behavior is one of the most important factors that affect the mechanical properties of Ni-Ti alloy. Cold work and heat treatment are also important variables to control the mechanical properties of Ni-Ti alloy and improve the lifetime of endodontic files<sup>21-23</sup>. Obviously, as heat treatment has an effect on
phase transformation behavior, it is very important to identify phases.

DSC is a powerful tool for materials characterization of Ni-Ti rotary instruments, providing direct information about the Ni-Ti phases present, which is not readily available from other analytical techniques. Therefore, in this study, DSC was used to detect the effects of heat treatment on phase transformation behavior. From the DSC results of this study, $\Delta f$ for groups 400 and 450 exceeded the body temperature of 37°C. This means that the alloy of these groups was a mixture of both austenite and martensite in the oral environment. Conversely, $\Delta f$ for group 500 was around 37°C, and $\Delta f$ for groups 300, 600 and the control group, meaning that, in the oral environment, these instruments consisted mainly of austenite. It was expected that these differences in phase composition would affect the bending properties.

**Bending properties**

A typical stress-strain curve of a Ni-Ti super-elastic alloy forms a hysteresis loop. The L/D ratio is equivalent to the elastic modulus of the Ni-Ti alloy, which is dependent on the component phases; the modulus of the martensitic phase is lower than that of the austenitic phase. Therefore, the flexibility within the elastic range is influenced by $M_s$ and $A_s$. From the bending results in this study, the L/D ratios of groups 400 and 450 were lower than those of the other groups at 37°C. This is because that the specimens in groups 400 and 450 contain a mixture of austenite and martensite, while the others consist mainly of austenite.

After the stress exceeds the elastic limit, the phase of the alloy changes by stress-induced martensitic transformation. Austenite is transformed to martensite during loading and returns to austenite when unloaded. In this study, the mechanical properties of the specimens within the super-elastic range are characterized in terms of the 2.0-mm deflection bending load, which, in turn, is dependent on the stress-induced martensitic transformation.

For groups 400, 450 and 500, the bending load values in the super-elastic range were less than that for the control group. A linear relationship exists between the stress and temperature; the martensitic transformation stress increases with decreasing $M_s$. $M_s$ values for groups 400 and 450 were significantly higher than that for the control, and the critical stress for inducing martensitic transformation was reduced. The austenitic phase is more stable at high temperature, meaning that a higher stress is required for stress-induced martensitic transformation. The difference in the transformation temperature is thought to be one of the reasons for the load values at the 2.0-mm constant deflection found for groups 400, 450 and 500 compared with that of the control group. For group 600, there is a small permanent residual strain after unloading, because the recrystallization takes place.

**Cyclic fatigue properties**

Two different fracture types have been reported for Ni-Ti endodontic instruments, and may be caused by different mechanisms: torsional load and flexural fatigue of the alloy. Torsional load is transferred into the instrument via friction against the canal wall. Torsional fracture typically occurs when an instrument tip is forced into a root canal that is smaller than the tip diameter; the tip locks into the canal, does not follow the speed of rotation, and breaks. In such a situation, the coronal fragment often shows plastic deformation. In contrast, fracture due to cyclic fatigue occurs when a metal is subjected to repeated cycles of tension and compression that cause its structure to break down. Cyclic fatigue often occurs unexpectedly, and may leave few signs of permanent deformation, mostly on the cross-sectional surface.

Parashos et al. reported that 70% of instrument separation that occurred during shaping could be attributed to flexural fatigue. Cyclic fatigue is the main mechanism leading to intracanal failure. In this study, a cyclic fatigue testing device designed by Jamleh et al. was used. For engine-driven Ni-Ti rotary instruments, the radius of curvature, angle of curvature, and instrument size are more important than the operating speed for predicting separation.

The cyclic fatigue testing was performed at constant deflection. Fatigue life is generally a strong function of stress. Increasing the stress decreases the fatigue life. From the bending test results of this study, instruments stress differed among groups under particular conditions of deflection. The deflection of 1.0 mm is probably within the limits of the elastic range, while that of 1.5 mm might be within the super-elastic range for these experimental conditions, judging from the load-deflection curves for each group (Fig. 3). So, cyclic fatigue resistance under R10 condition could be dependent on the component phases. Therefore, the NCFs of groups 400, 450 and 500 were greater than that of group 600 under the R10 condition, because the L/D ratios or the elastic moduli of groups 400, 450 and 500 were significantly lower than that of group 600. However, resistance to cyclic fatigue under the R5 condition could be reliant on the stress-induced martensitic transformation. The NCFs of groups 400, 450 and 500 were significantly greater than that of group 600 under R5 condition because these groups maintain their martensitic state under these conditions.

Ni-Ti instruments on the market have different compositions, manufacturing processes and design aspects (including tip sizing, taper, cross section, helical angle and pitch). In this study, conical specimens were used to investigate the influence of heat treatment, because it was difficult to exclude the effects of the differences between products on the market. In the current study, a newly developed Ni-Ti rotary instrument produced through a proprietary thermomechanical process was reported to have more flexibility and cyclic fatigue resistance than conventional super-elastic Ni-Ti instruments. The results of this.
study coincide with those of Gao et al.\textsuperscript{20}. Therefore, we have confirmed that correct heat treatment may make specimens more flexible and resistant to cyclic fatigue.

CONCLUSIONS

The influences of heat treatment on the mechanical properties of Ni-Ti file materials were investigated using DSC, a cantilever-bending test and a cyclic fatigue test. The following conclusions were drawn:

1. The transformation temperature was changed by heat treatment, and this change in the transformation temperature was related to the heat treatment temperature.

2. The L/D ratios of groups 400 and 450 were lower than that of the control group. The bending load values at 2.0 mm of groups 400, 450 and 500 were lower than that of the control group. The bending properties of Ni-Ti endodontic instruments may be closely related to the transformation behavior.

3. Group 500 displayed the maximum NCF under R10 conditions. Under R5 conditions, the NCFs of groups 400, 450 and 500 were greater than that of the control group.

4. The change in flexibility with heat treatment may have an effect on the cyclic fatigue properties of Ni-Ti endodontic instruments. Ni-Ti endodontic instruments with great flexibility may exhibit more resistance to cyclic fatigue.

REFERENCES