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Microstructural effects on Ni-Ti endodontic instruments failure

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Abstract. Endodontic treatments consist to eliminate the vascular nervous system of the tooth. The objectives of this treatment are adequate cleaning and shaping the root canals of the tooth. The endodontic treatment is essential to the success of prosthetics therapy and the lifetime of the tooth. The difficulties of endodontic treatments lie in the abruptness of canal curvature. Ni-Ti endodontic instruments were introduced to facilitate instrumentation of curved canals. They are superelastic and flex far more than stainless-steel instruments. Despite the increased flexibility we can observe unexpected fractures of these Ni-Ti files. The purpose of this work is to understand the process history on fracture life. Our results are based on microstructural and mechanical investigations of Ni-Ti engine-driven rotary files : X-ray diffraction, SEM, DSC, microhardness and bending tests. Thus, and as we expected, endodontic files are very work-hardened: there is a high density of defects in the alloy, which will impede the phase transformation. DSC : the phase transformation A/R-Phase is predominant, the martensitic transformation is difficult to observe. The microvickers hardness confirms these observations (dislocations and precipitates). The X-rays show that the experimental peaks are broad, which is typical of a distorted lattice. Morover, machining resulted in the work hardening of files. Some thermal treatments are involved in promoting some changes in the mechanical properties and transformation characteristics. Annealing around 400°C shows good results : the recovery allows a compromise between an adequate density of defects to see the R-Phase germination and a low density to limit the brittleness of these instruments. The surface state of the endodontic files is an important factor for failures and fractures initiation. In these applications, it is very critical to predict the service life based on the theoretical modeling.

1. INTRODUCTION

The NiTi alloys have interesting applications in medicine. Two special applications are found in dentistry field concerning orthodontic and endodontic therapies. Endodontic treatments consist to eliminate the vascular nervous system of the tooth. The endodontic instruments are used to prepare the tooth canal for restorative procedures. They are little conical files, which are employed to erode the canal and enlarge its diameter maintaining the original shape. Difficult endodontic treatments are related to abruptness of the canal curvature ; the risk with traditional files (stainless-steel) is plastic deformation and fracture due to stress and strain in the canals. Consequently, new endodontic instruments have been developed in order to minimize the breakage risk. Ni-Ti instruments were introduced to facilitate instrumentation of curved canals. These instruments are superelastic and will flex far more (10-20 times more) than stainless-steel instruments before exceeding their elastic limit (1, 2). Despite this increased flexibility, many authors have reported unexpected fractures (3).

The SE (superelasticity) nature of NiTi has been attributed to a reversible austenite to martensite transformation. It is believed that austenite is transformed into martensite during loading and reverts back to austenite when unloaded. The transformation is reversible during clinical use because SE alloys have a TTR (transition temperature range) lower than mouth temperature. The TTR of NiTi is affected by the composition and the manufacturing techniques of the alloy (4).

Various heat treatments can modify SE behaviour. Miyazaki found SE to be greatly dependant on the thermal history of the alloy (5). We performed some thermal treatments which are involved in promoting some changes in the mechanical properties and transformation characteristics. These properties can be modified by high dislocations density and/or fine dispersion of particules.

The purpose of this work is to understand the process history on fracture life. The instruments are studied by microstructural and mechanical investigations.

2. MATERIALS AND METHODS

2.1 Materials

Our investigations concern the endodontic engine-driven rotary instruments produced by Maillefer Instrument SA (Ballaigues, Switzerland) and by Micromega (Besançon, France), respectively known with trade names "ProFile" and "Hero". The studied files have a 25 mm length, a taper ranging between 0.02,

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0.04 and 0.06 mm per mm length and sizes 20 to 40, representing the diameter of the tip base of the file, given in hundredth of millimetre.

Specimens were cut to separate working or active part of the file and the inactive part using a low-speed diamond saw. Several samples were chosen: new instruments or instruments that have been used in clinical conditions (12 or 18 root canals and about 5 or 10 sterilizations).

2.2 Thermal treatments

Different thermal treatments have been investigated. The heat treatments consisted of an annealing at 350-400-450-510-600-700 degrees Celsius in salt baths during 10 min, and at 600 and 700 °C during 15 min with the same process, and subsequent water quench. The influence of different heat-treatments on NiTi alloys was investigated by DSC-measurements, microhardness tests and bending tests.

2.3 Methodologies

Differential Scanning Calorimetry.

The transformation temperatures were determined by DSC (Mettler 30/TA 4000). The mass of the samples for DSC measurements were 18 mg, heating and cooling rates were 5°C/min. Specimens were placed in Al crucible pan with nitrogen gas flow environment. The thermograms were carried out in a range of temperature of + 60°C and -120°C.

X-Rays Diffractions.

A Philips diffractometer was used to detect the present phases at room temperature. The wavelength of the Cu Kα radiation used in this study is 0,154051 nm.

Microhardness.

For mechanical characterization, the Vickers Microhardness was measured with a weight less than 1,96 N. These tests were carried out at room temperature and only on the inactive part of the file (without machining). As the number of samples was small (10), the analysis of our results will be performed with a Kruskal-Wallis test.

SEM.

Size 20 Hero and Profile 0.04 and 0.06 taper were observed by Scanning Electron Microscope in order to study the surface states. A JEOL T330 was used and the EDS analysis was performed on a Tracor TN 5500.

Bending tests.

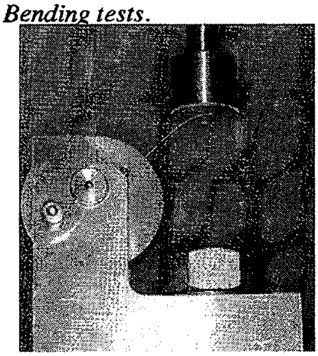

Figure 1 :Bending testing machine.

To perform bending tests, we arranged a bending testing machine (Fig: 1). All the instruments are loaded with the same displacement and the force corresponding is calculated by the cell (100 N). The loading and the unloading are performed in same conditions.

On clinical conditions, files are mostly solicited on bending; we tried with our machine to reproduce this sort of solicitations.

New instruments, instruments used in clinic, instruments that have been heat treated are included for this mechanical test. We will obtain informations about the "elastic" behaviour (flexibility) of files and about heat treatments and clinical use. The results are discussed only in a qualitative analysis and not a quantitative analysis, because of the shape of instruments (range and machining design) which prevents any calculation.

3. RESULTS

3.1 DSC

The transformation process can easily be recorded by measuring the transformation latent heat released/absorbed to/from the surroundings. Fig.2 displays the DSC curves for different conicities of Hero. It shows one distinct transformation on cooling, austenite (A) to R-phase (R). On heating, two peaks are obtained (two-step transformation M->R and R->A).

The peaks are better and better defined with increasing of conicity. The ProFile samples show the same characteristics but all peaks are less defined.

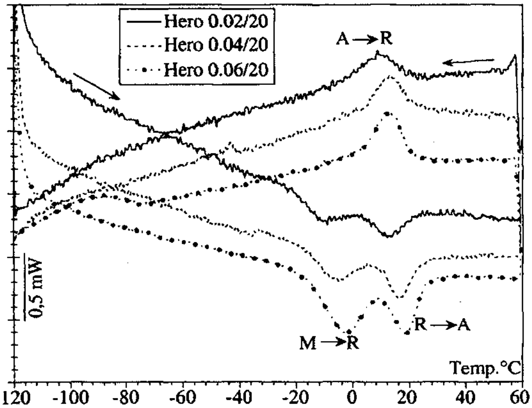

Figure 2 : Comparison between different conicity.

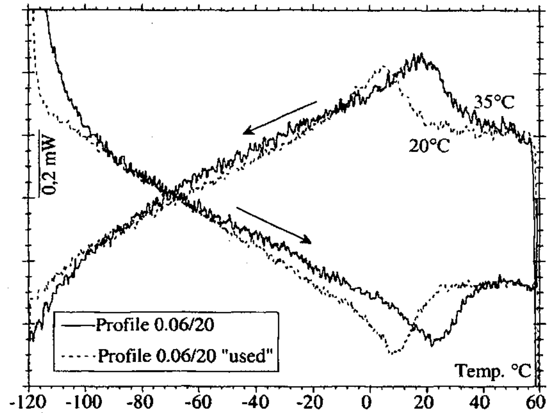

Figure 3 : Comparison between new and used Profile.

Figure 3 shows the DSC curves for new ProFile and ProFile that have been used in clinical conditions (12 root canals and about 10 sterilizations). In the case of the new file, one peak is obtained on cooling and on heating (A -> R). However, when the file is used,the same peaks are observed but the TTR shifts to lower temperatures (about 15 °C lower).

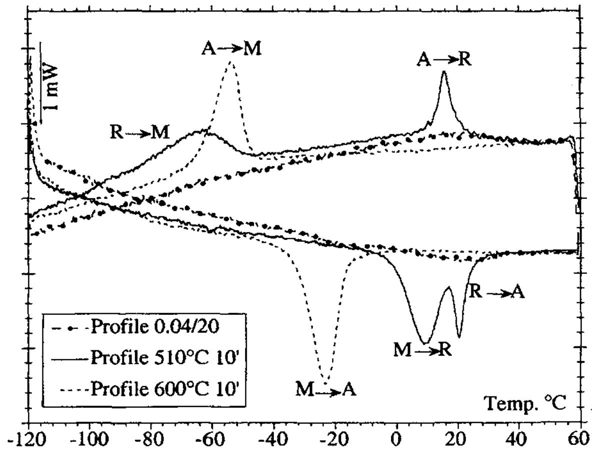

Figure 4 : DSC curves after heat treatments.

After heat treatments, the samples show two different types of transformations courses during DSC-measurements (Fig. 4). The peaks intensity and position change with varying annealing conditions. After heat treatments at 510°C, two peaks are found during cooling process and two peaks during heating (A-> R, R-> M). The 600°C thermal treatment yields a transformation behaviour with one peak during cooling and during heating. When the annealing temperature is above the recrystallization temperature 600°C, only one exothermic peak can be found during cooling (A-> M). We have the direct and reverse A-> M transformation, the R-phase transformation can not be detected. This thermal treatment shifts the martensite transformation to lower temperatures.

3.2 X-rays diffraction

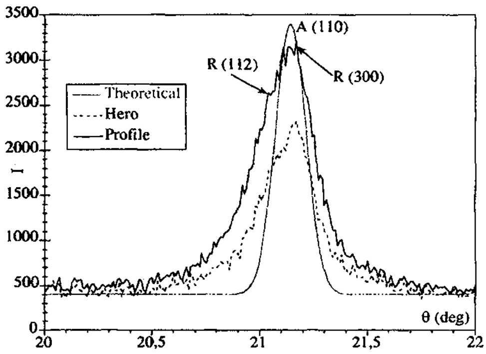

Figure 5 : Enlargement of the peak (110).

X-ray diffraction of Hero and Profile files showed that Hero is fully austenitic and Profile is biphased but almost Austenitic.

The XRD scans of all specimens show a (110) texture. Peak width is an indication of cold work and lattice distortion.

From an enlargement of the peak (110) (Fig. 5), it is apparent that the experimental spectrum lines are extended; this shows that the lattice has been distorted and therefore that the alloys are work hardened (6).

3.3 Microhardness

This test compares data relating to each instrument and shows a statistically significant difference among these instruments (p<0,005). Table 1 shows the average value for each instrument :

For size 20 Hero 0.06 taper and size 20 ProFile 0.06 taper, there was no statistically significant difference (p=0,0179). After comparison of two types of heat treatments (superior to 550°C and inferior to 550°C); no significant difference was found into each type but there is a statistical difference among each group (p=0,0001).

Table 1 : Microhardness results. HV = Hardness Vickers.

Endodontics files	*Average (HV)*
Hero 6 20	421
Profile 6 20	475.2
ProFile 4 20 – 350°C 10'	407.2
ProFile 4 20 – 400°C 10'	420
ProFile 4 20 – 450°C 10'	401.6
ProFile 4 20 – 510°C 10'	372.4
ProFile 4 20 – 600°C 10'	258
ProFile 4 20 – 600°C 15'	258
ProFile 4 20 – 700°C 10'	254.4
ProFile 4 20 – 700°C 15'	254.2

An other difference appears between size 20 Profile 0.06 taper and size 20 Profile 0.04 taper which were heat treated at temperature inferior to 550°C (p=0,0013).
Our samples, before any use, have a hardness above 400 HV: these samples are already work hardened, probably with precipitates. The samples annealed at a temperature below 600°C have certainly a smaller density of defects (dislocations) ; for temperatures above 600°C, the alloy recrystallizes and the precipitates partially disapear.

3.4 SEM

For Scanning Electron Microscopic examination, the endodontic files, are bent, to generate stress-strain on the most curvated part of the file. For both instruments, SEM micrographs of the curved body region of size 20 taper 0.06 Profile and Hero show significant machining marks along the faces of the flutes.

Figure 6 : A size 20 Hero taper 0.06, striation patterns observed on curved body region.

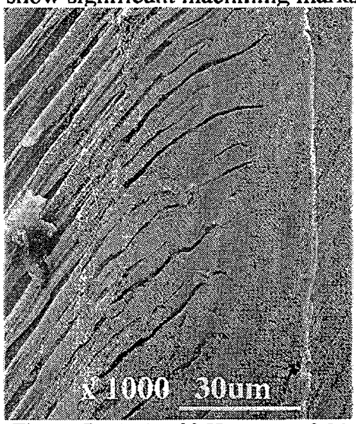

Figure 7 : A size 20 Hero taper 0.06, "used", cracks appear on cutting edge.

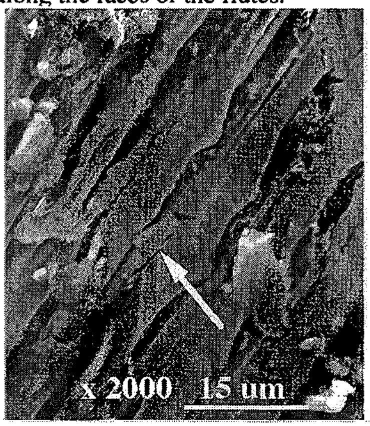

Figure 8 : A size 20 Hero taper 0.06, "used", the head of the arrow lies at a microcrak.

For the curved body region of size 20 Hero 0.06 taper, observed after clinical utilisation (10 root canals and 4 sterilization cycles), the cutting edges and ridges show irregularities and cracks. These cracks are easily identifiable because of the curvature of the instrument (Fig. 7). They look like fatigue fracture surfaces.
At a higher magnification (Fig. 8), on the top of the curvature, real sinuous-shape cracks appear. We can also see scraps from machining.

3.5 Bending test

At first and until 3mm of strain, only the tip of the instrument bends. Then between 3 and 6mm, the curvature is now in the half-way part of the file. Finally, above 6mm, the part which has the maximum section, near the handle, becomes deformed at its turn.

The stiffness increases with the conicity (Fig. 9). For taper 0.06, tip of Hero is less flexible than ProFile but near the handle, the behaviour is quite similar.
For taper 0.04, the only difference is the increased rigidity of ProFile near the handle.

We can not notice any difference between new instrument and file that has been used in straight canals (Fig. 10). But, when abruptness of canal curvature increases, the stiffness of the "used" file increases after each utilisation.

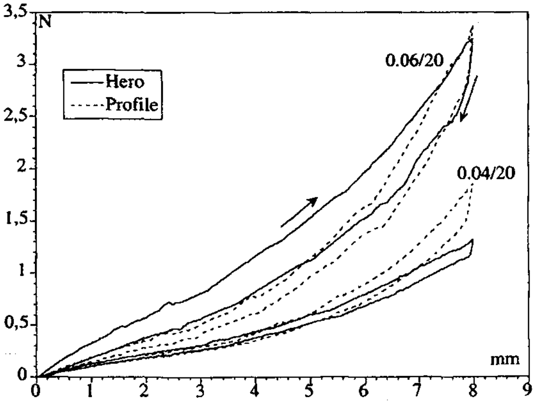

Figure 9 : Comparison between Hero and ProFile for two tapers.

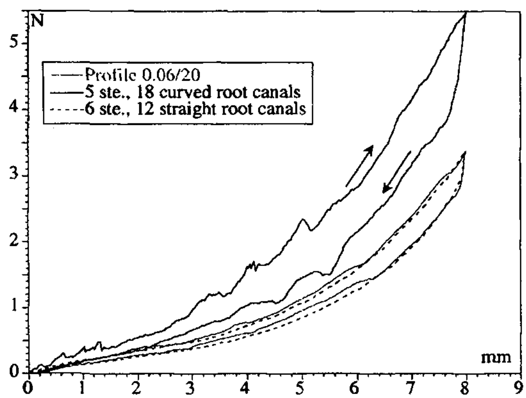

Figure 10 : Comparison between new and "used" ProFile 0.06/20.

Figure 11 (a and b) demonstrates that the annealing conditions have strongly affected the stress-strain behaviour. For heat treatments above recrystallization temperature, the stiffness of the instruments increase. On the other hand, results show that after annealing at a temperature below recrystallization, the specimens generally show an increased flexibility.

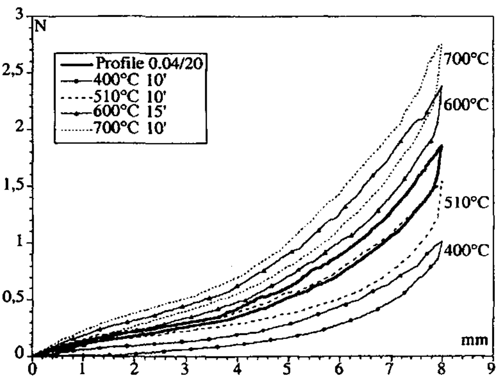

Figure 11a : Bending curves for various annealing conditions.

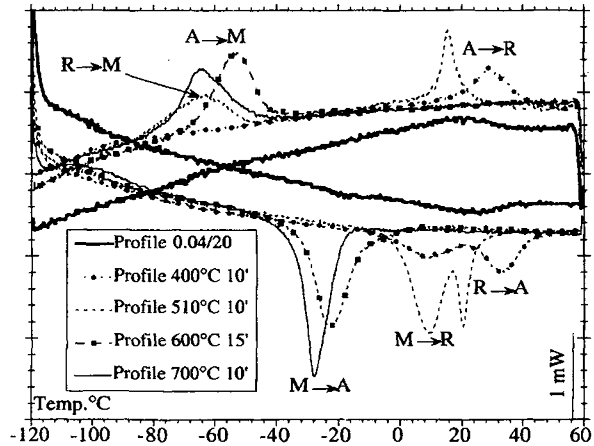

Figure 11b : DSC thermograms for various annealing conditions.

4. DISCUSSION - CONCLUSION

Mechanical fatigue of Ni-Ti alloys was studied : fatigue crack growth rates were measured and formed to be lower than predicted from the phenomenological law relating growth rates to the elastic modulus (7). A deviation of a factor of 3 is observed with conventionnal metals or alloys. This decrease in normalized crack growth rate may be a consequence of reversible martensitic deformation processes leading to less accumulation of damage per cycle compared with more conventional materials.

Our results with DSC, XRD and microhardness show that new specimens are significantly work-hardened : microhardness mesure is twice than a sample fully recrystallized (8). Moreover machining marks and cracks on the surface observed by SEM contribute largely to fatigue failure by crack propagation process. The crack nucleation stage is much facilitated by the high density of surface defects. Manufacture of Ni-Ti alloys by machining in endodontic instruments promote work hardening and create surface defects.

These first observations (microstructure and surface defects) allow to explain by now unexpected fractures reported in litterature (3).

Dislocations present in the matrix influence the mechanical properties, internal stresses are a negative factor to the mobility of martensite interfaces (9). Moreover, DSC thermograms (Fig. 3) and bending curves (Fig. 10) of "used" instruments (abrupt curvatures) show an increased density of dislocations. Indeed, the shift of the TTR to lower temperature impedes the phase transformation, instruments become stiff. It appears that the abruptness of the curvature of the root canals (stress field) is the essential parameter, and that the numbers of root canals treated is less important for the increase of brittleness. Although manufacturers advise not to use each file on more than 10-12 root canals.

Heat treatments are known to influence mechanical properties and various phase transformation temperature of Ni-Ti SMA. Two annealing temperature ranges can be distinguished. In the first case, annealing temperatures about 600°C (recovery) show two-step transformations (A <-> R <-> M) ; in the second case, when annealing temperatures are above 600°C (recrystallization) we can observe a direct martensitic transformation. With the beginning recrystallization (600 and 700°C), R-phase transformation can not be detected. It can be presumed that the decreasing dislocation density and any precipitation stress fields are not able to initiate the R-phase transformation. At this point microhardness reaches its minimum average value (Table 1). The dissolution of Ni-rich precipitates increase Ni-content in the matrix and shift the TTR to lower temperature. Thus the stiffness is much more important. For clinical applications, these heat treatments are not required.

Between the cold worked initial state and the 400°C heat treated, the average values of microhardness are similar. According to result from Nishida and al. (10) Ni-rich precipitations (Ni_4Ti_3, $Ni_{14}Ti_{11}$) may have occur in spite of a slight decrease in dislocation density. Furthermore, the internal stress fields around the precipitates and dislocations favour the R-phase nucleation. The TTR of R-phase transformation is nearer than the temperature of clinical utilisation. The best flexibility is observed on bending curves for this heat treatment (Fig. 11a).

In these cold-worked files, the high dislocation density influences the reorientation processes and the crack growth : the instruments become brittle. As concerns superelasticity, with cycling, reorientation of the martensite under stress leads to gradual defect accumulation and it might be expected that these dislocations are generated at the interface between different martensite colonies. In clinical conditions, the curve of canals distorts the endodontic instruments; cyclic fatigue is caused by repeated tensile-compressive stress. The maximum of this stress is in the surface of the curve. Crack nucleation and propagation stages appear mostly on the half of the instrument which is in tension (outside of the curve).

Some suggestions could be proposed to improve the lifetime of endodontic files: apply a thermal treatments at about 400°C (recovery) before machining to decrease the work-hardening of the alloy ; choose machining conditions adapted to this NiTi shape memory alloy ; an electropolishing procedure could be used by the manufacturer to reduce the machining dammage on the file surface.

Acknowledgement

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