

# Bending Properties of a New Nickel-Titanium Alloy with a Lower Percent by Weight of Nickel

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## Abstract

**Introduction:** The aim of the present study was to evaluate the bending properties of Hyflex instruments, which exhibit a lower percent in weight of nickel (52 Ni %wt) and compare them with other commercially available nickel-titanium (NiTi) rotary instruments.

**Methods:** Ten instruments with tip size 25, 0.06 taper of each of the following NiTi rotary instrumentation techniques were selected for the study: Hyflex, EndoSequence, ProFile, Hero, and Flexmasters. All instruments from each group were tested for stiffness by comparing their bending moment when they attained a 45-degree bend. Experimental procedures strictly followed testing methodology described in ISO 3630-1. All data were recorded and subjected to statistical evaluation by using analysis of variance test. Statistical significance was set at  $P < .05$ . **Results:** Statistical analysis of the data revealed that Hyflex files were found to be the most flexible instruments, with a significant difference ( $P < .05$ ) in comparison with the other instruments. Among the other files, a significant difference has been reported for EndoSequence instruments compared with ProFile, Hero, and FlexMaster ( $P < .05$ ), whereas no significant differences have been reported among those 3 files ( $P > .05$ ). **Conclusions:** Results of the present study have illustrated an increased flexibility of the new NiTi alloy over conventional NiTi alloy, and they highlight the potential of the new manufacturing process. (*J Endod* 2011; ■:1–3)

## Key Words

Endodontic instruments, flexibility, nickel-titanium, thermal treatments

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doi:10.1016/j.joen.2011.05.023

One of the major innovations in endodontics has been the introduction of nickel-titanium (NiTi) alloy to manufacture root canal instruments. This is mainly because of the superelasticity of the NiTi alloy, which provides increased flexibility and allows the instruments to effectively follow the original path of the root canal (1). Nitinol is a simple binary mixture of nickel and titanium at about 50 atomic percent each (about 55% by weight of nickel). However, subtle adjustments in the ratio of the 2 elements make a large difference in the properties, particularly the transformation temperatures, ie, the temperatures at which the crystal structure changes from austenite to martensite or vice versa. The sensitivity of the transformation temperature to composition is so great that chemistry is not used to specify the alloy. Instead, transformation temperature is the most accurate means to specify the alloy. The temperature most frequently specified for the finished product is the active austenite finish temperature, Active A(f). This is determined by differential scanning calorimetry (DSC) by using American Society for Testing and Materials (ASTM) F 2082. Typical tolerances for Active A(f) are  $\pm 5^\circ\text{C}$ .

For shape memory NiTi alloys, the Active A(f) determines the completion of the shape recovery transformation on heating. For superelastic NiTi alloys, the Active A(f) must be below the product use temperature. A superelastic material will remain superelastic up to a temperature from the Active A(f) to a temperature about  $50^\circ\text{C}$  above Active A(f). Therefore, a material with an Active A(f) of about  $15^\circ\text{C}$  will exhibit good superelasticity up to about  $65^\circ\text{C}$ . However, the greatest ability to recover occurs close to A(f), as shown by Duerig et al (2).

The transformation temperatures change because of mechanical processing and annealing; therefore, the Active A(f) of NiTi rotary instruments will be different than the transformation temperature of the original ingot. In a recent article, Hou et al (3) showed that a different manufacturing process, including a proprietary heat treatment, produced instruments (Twisted File; SybronEndo, Orange, CA) with significantly different Active A(f) and, consequently, significantly different bending properties. In previous studies, it has been shown that files produced with this new manufacturing technique resulted in instruments having enhanced mechanical properties and resistance to cyclic fatigue, compared with instruments manufactured with a traditional grinding process (4–6).

Possible strategies to increase efficiency and safety of NiTi rotary files include an improvement in the manufacturing process or the use of new alloys that provide superior mechanical properties. Heat treatments are performed during the production of the NiTi ingot but can also be performed by endodontic manufacturers before, during, and after the manufacturing of the NiTi rotary instruments. Unfortunately, all these heat treatments are usually proprietary, and they are not disclosed by manufacturers. According to Zinelis et al (7), this explains, contrary to other dental and biomedical alloys, why the mechanical properties and transformation temperatures of NiTi alloys are usually dominated by the hidden thermomechanical history rather than the elemental composition. One of the very few exceptions, however, can be Hyflex instruments (Coltene-Whaledent, Allstetten, Switzerland), which exhibit a lower percent in weight of nickel (52 Ni %wt) than the common 54.5–57 Ni %wt shown by the great majority of commercially available NiTi rotary instruments (7). Hyflex is a new NiTi rotary file for root canal preparation that has been commercialized since 2011. Hyflex NiTi Files have been produced by an innovative methodology (patent pending) that uses a unique process that controls

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the material's memory (a complex heating and cooling treatment). According to many authors, such compositional deviations between 54 and 57 Ni %wt, probably attributed to raw material variations during manufacturing, did not seem to affect the mechanical properties of NiTi instruments (8–11). However, none of these studies evaluated Hyflex instruments, the only one with a 52 Ni %wt. Therefore, the aim of the present study was to evaluate bending properties of the Hyflex instruments and compare them with other commercially available NiTi rotary instruments.

### Materials and Methods

Ten instruments with tip size 25 and 0.06 taper of each of the following NiTi rotary instrumentation techniques were selected for the study: (1) Hyflex (Coltene-Whaledent, Allstetten, Switzerland), (2) EndoSequence (Brasseler, Savannah, GA), (3) ProFile (Dentsply-Maillefer, Baillagues, Switzerland), (4) Hero (MicroMega, Besancon, France), and (5) Flexmaster (VDW, Munchen, Germany).

All instruments from each group were tested for stiffness by comparing their bending moment when they attained a 45-degree bend. Experimental procedures strictly followed testing methodology described in ISO 3630-1 (12) by using a computerized device (Fig. 1).

Testing device (MAGTROL FTS-100, Buffalo, NY) and test procedures were in accordance with specifications described for root canal files. Three millimeters of the tip of each instrument was clamped in a chuck connected to a digital torque meter memocouple and to a strip chart for data recording. The amplifier was set at an angular deflection of 45 degrees, at which point the test stopped automatically. The bending moment was then measured and recorded by the memocouple, and the value was read directly on the strip chart.

All instruments had been previously examined under a measuring microscope (MS2 Walter Uhl, Asslar, Germany) at D3 (3 mm from the file tip) and D16 (16 mm from the file tip) to ensure uniformity of dimensions (according to the tolerance indicated by ISO 3630-1) and under a stereomicroscope (Karl Kaps GmbH & Co KG, Asslar, Germany) at 20 $\times$  magnification to ensure uniformity of cutting flutes and defect-free surfaces. All defective instruments were eliminated from the study and substituted with new ones.

All data were recorded and subjected to statistical evaluation with analysis of variance test. Statistical significance was set at  $P < .05$ .

### Results

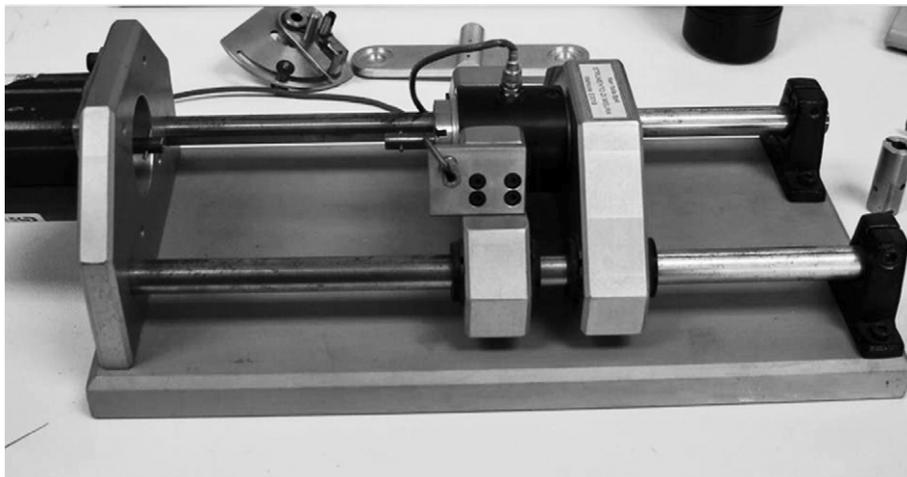
Mean values and standard deviation for each group of instruments are shown in Table 1. The higher the value, the more rigid were the instruments. Statistical analysis of the data revealed that Hyflex files were found to be the most flexible instruments, with a significant difference ( $P < .05$ ) in comparison with the other instruments. Among the other files, a significant difference was found for EndoSequence instruments compared with ProFile, Hero, and FlexMaster ( $P < .05$ ), whereas no significant differences have been reported among the latter 3 files ( $P > .05$ ).

### Discussion

Thermal treatment of NiTi alloys for endodontic use is a new field of research, and little information is currently available. Thermal treatment of the alloy is known to produce a better arrangement of the crystal structure, thus leading to improved flexibility (superelastic behavior), and also changes in the percentage of phases (a different grain structure) of the alloy, thus leading to improved resistance or plastic behavior. Patented, proprietary processes are highly influenced by temperature and time intervals, and each small change makes every single manufacturing process unique.

Energy-dispersive x-ray analysis performed by Zinelis et al (7) showed that Hyflex instruments had a lower percent in weight of nickel (52.1 Ni %wt) than the other tested NiTi rotary instruments. In fact, the values for FlexMaster, EndoSequence, Hero, and ProFile were 56.2, 56.0, 55.4, and 54.6 Ni %wt, respectively. These values were close to those (56 wt% Ni, 44 wt% Ti) reported previously (1, 13–17). These values are within the nominal composition range specified in the ASTM standards for wrought NiTi alloys used in medical devices and surgical implants (F2063-05 2005).

Results from the present study showed that Hyflex instruments were the most flexible instruments, with a significant improvement in flexibility over the other tested commercially available instruments (Table 1). Despite the fact that the flexibility is influenced by instrument's design, such a great improvement is probably related to the proprietary manufacturing process (which is not disclosed by the manufacturer) and/or the different percent in weight of nickel of the Hyflex instruments, because they have a cross-sectional design very similar to EndoSequence. Because the Hyflex instruments are new to the market and there is no older similar files that were manufactured with



**Figure 1.** The testing device used in the present study for the stiffness test.

**TABLE 1.** Mean Values  $\pm$  Standard Deviation of the Values Obtained in the Stiffness Test

Instrument	g/cm
Hyflex	14.46 $\pm$ 2.7 <sup>a</sup>
EndoSequence	46.01 $\pm$ 3.9 <sup>b</sup>
ProFile	64.18 $\pm$ 4.2 <sup>c</sup>
Hero	67.11 $\pm$ 5.7 <sup>c</sup>
FlexMaster	68.39 $\pm$ 4.9 <sup>c</sup>

The lower the value, the more flexible the instrument is. Different superscript letters indicate statistically significant differences.

a traditional grinding process without any thermal treatment or with a similar manufacturing process (with proprietary heating and cooling processes) but with a 55% Ni wt, it is not easy to determine whether the improvement in the flexibility is due to the unique composition, the property thermal proportion, or both. Several studies have shown that phase transformation behavior has an impact on the mechanical properties of NiTi instruments including flexibility (18–22), and the former is easily influenced by factors including chemical composition, heat treatment, and manufacturing processes (1, 23). Furthermore, Zinelis et al (7) reported that Hyflex compositional deviations did not seem to affect the mechanical properties of the NiTi instruments, and that the thermomechanical history has a much more crucial effect on the final mechanical strength (8–11). It can be postulated that the proprietary thermal processing plays an important role in increasing flexibility of Hyflex instruments when compared with the other tested instruments, which have a traditional manufacturing process with no property thermal treatments and, consequently, different thermomechanical history. The potential role of different percentage in weight of nickel of the Hyflex instruments still remains uncertain. However, because this compositional deviation was attributed to raw material variations during manufacturing and thermal processing (7), we might consider it as a result of a unique thermomechanical history of the alloy. Therefore, it is difficult to determine whether there is a crucial effect of a single factor or, more likely, a combination of different correlated factors in determining the final mechanical properties of Hyflex instruments.

On the contrary, the improvement in flexibility shown by EndoSequence compared with Hero, FlexMaster, and Profile, all manufactured with the same grinding process and with similar energy-dispersive x-ray patterns, is mainly related to the unique shape of the EndoSequence instruments, with a smaller inner core and alternate flute design.

During the last decade several changes in cross-sectional and flute design have been introduced to increase flexibility of greater tapered NiTi rotary instruments, which are intended to be used in apical portion of curved root canals (ie, size 25/0.06), but only small improvements were achieved. Therefore, a significant improvement in the flexibility of the alloy should be highly beneficial, providing NiTi instruments of greater taper with a superior ability to negotiate curved canals, to reduce the tendency of iatrogenic errors, and to allow dimensionally adequate apical preparations of curved canals while maintaining the original path.

Flexibility is one of the most important properties of NiTi rotary instruments, but it cannot be the only characteristic on which a choice among different instruments is based. Moreover, when new root canal instruments are produced with innovative manufacturing process or alloy properties that differ markedly from conventional files, several characteristics (ie, torsional and flexural resistance, cutting ability, wear, etc) need to be investigated and tested to allow an efficient and safe clinical usage. Therefore, more tests are needed to fully evaluate advantages and, eventually, disadvantages of instruments produced with an alloy that exhibits

a lower percent in weight of nickel (52 Ni %wt) than the common 54.5–57 Ni %wt shown by the great majority of commercially available NiTi rotary instruments.

Results of the present study have illustrated an increased flexibility of the new NiTi alloy over conventional NiTi alloy, and they highlight the potential of the new manufacturing process.

## Acknowledgments

*The authors deny any conflicts of interest related to this study.*

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