Original Article

# A Comparative Study of Four different Contemporary NiTi Rotary Endodontic Files by Metallurgical and Mechanical Analysis with Energy-dispersive X-ray Spectrophotometry with FE-SEM and Cyclic Fatigue Resistance Evaluation

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## Abstract

**Aims:** The aims of this study were to evaluate the various properties of nickel-titanium (NiTi) endodontic rotary instruments and to assess the influence and clinical relevance of their metallurgy properties. **Materials and Methods:** Four different commercially available brands of NiTi endodontic rotary instruments (Protaper Gold, Hyflex CM, HyFlex EDM, Neolix Neo NiTi) are to be evaluated. These instruments were subjected to cyclic fatigue resistance testing in artificial metallic canals until fracture occurred. NiTi alloy properties were investigated by energy-dispersive spectroscopy. Field emission scanning electron microscopy analysis was conducted on new files to examine surface characteristics. **Results:** Analysis of variance testing revealed significant differences ($P < 0.05$) among the groups. Hyflex EDM Files presented the highest fatigue resistance. **Conclusion and Clinical Relevance:** All samples showed surface irregularities. Hyflex EDM instruments demonstrated the highest number of cycles to failure values in all the tested instruments. Hyflex EDM achieved the highest resistance to cyclic fatigue among the tested files.

**Keywords:** Cyclic fatigue, Energy-dispersive spectroscopy, Field emission scanning electron microscopy, Metallurgical analysis.

## Introduction

Nickel-titanium (NiTi) alloy was introduced in endodontics in the year 1988.[1] It exhibits shape memory and superelastic properties. These properties allow for improved access to curved root canals during the canal preparation. The success behind the NiTi alloy resides in its biological and mechanical properties that make the NiTi alloy unique and particularly suitable for the manufacturing of endodontic rotary instruments: biocompatibility,[2] corrosion resistance,[3] shape memory effect, and superelasticity. The past two properties have a key role in determining the mechanical behavior of NiTi rotary instruments.

- The first one, the shape memory effect, allows NiTi instruments to "memorize" a certain form and return by heating to its original form thanks to the phase transition between two crystallographic phases.
- The second one, the superelasticity, allows NiTi alloy, when mechanically loaded, to be reversibly deformed to very high strains (up to 8%) by the creation of a stress-induced phase, called stress-induced martensite (SIM), which takes place when the ambient temperature is above the so-called austenite – finish temperature of the material. This occurs because NiTi alloy is able to undergo a non-diffusive transformation of the lattice structure into a martensitic phase when suitably stressed. Such stress-induced martensitic (SIM) transformation is reversible, and hence, the material exhibits an unusually large elastic range and is able to recover from a much higher strain than stainless steel can withstand without breaking. This special mechanical property is called superelasticity. This special

**Access this article online**

**Website:** www.jrad.co.in

**DOI:** https://doi.org/10.53064/jrad.2023.14.1.307

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**Received:** Oct. 19, 2022; **Accepted:** Nov. 08, 2022; **Published:** Dec. 15, 2022



**How to cite this article:** Azad A, Chokshi S. A Comparative Study of Four different Contemporary NiTi Rotary Endodontic Files by Metallurgical and Mechanical Analysis with Energy-dispersive X-ray Spectrophotometry with FE-SEM and Cyclic Fatigue Resistance Evaluation. J Res Adv Dent 2023;14(1):21-24.

property manifests as an enhanced elasticity of the NiTi alloy, allowing the material to recover after large strains. However, when the load is removed, the new SIM becomes unstable and the NiTi regains its original shape.[4]

Although literature regarding the mechanical properties of traditional shape memory rotary endodontic instruments is available, still limited information is provided regarding the mechanical and metallurgical properties of different contemporary endodontic instruments with their clinical correlation and significance which can be evaluated by mechanical tests such as cyclic fatigue resistance together with analyzing the compostion of the metal alloy.

### Aim

The aim of this study is to evaluate the elemental composition and microstructure and correlates it with mechanical properties of NiTi alloy of contemporary rotary endodontic instruments, to assess the relevance and clinical significance of their metallurgy properties.

## Materials and Methods

Forty different brands of contemporary NiTi rotary endodontic instrument were evaluated for the study, which were as following:-

1. Hyflex CM (25/0.06)
2. ProTaper Gold (PTG) (F2 - 25/0.08)
3. Hyflex EDM (OneFile - 25/0.08)
4. NeoLix NeoNiTi (A1 - 25/0.06).

Unused brand new rotary files were evaluated for the elemental composition and surface microstructure with the help of field emission scanning electron microscopy (FE-SEM) and energy-dispersive spectroscopy (EDX) after which cyclic fatigue test was conducted to check the flexural resistance of these files, where $n = 10$.

### Mechanical investigations

Instrument separation during canal preparation can be broadly divided into two types:

- Cyclic fatigue fracture
- Torsional fatigue fracture.

In the present study, cyclic fatigue resistance with energy-dispersive spectroscopy (EDS) was evaluated. In this study, ethical clearance was not required, as none of the samples had any human or biological tissue requirement or intervention. All the test were analyzed with engineering laboratory equipments.

#### *Cyclic fatigue*

Cyclic fatigue occurs when a metal is subjected to repeated cycles of tension and compression that cause its structure to break down, ultimately leading to fracture. Rotary instruments from each manufacturer were tested for cyclic fatigue resistance by a customized testing device. The device consists of a main frame to which a metallic support is connected for the placement of electric handpiece, and a stainless steel block containing the artificial canals. To prevent the instruments from slipping out and to allow for observation of the instruments, the artificial canals were covered with glass. The electric handpiece was mounted on this cyclic fatigue device to allow precise and reproducible placement of each instrument inside the artificial canal.

The artificial canal was manufactured by reproducing an instrument's size and taper. It provided the instrument with a suitable simulated root canal with a 60° angle of curvature and 5 mm radius of curvature. The instruments were rotated at a constant speed of 300 rotations per minute (rpm) using a 16:1 reduction handpiece (Contra angle handpiece, Neolix, France), powered by a torque controlled electric Endomotor (Endoman-E, Neolix, France). Torque was set at 2 N/cm. All instruments were rotated until fracture occurred. The time to fracture (TTF) was recorded visually with a 1/100-second chronometer. The TTF was multiplied by the number of rpm, to obtain the number of cycles to failure (NCF) for each instrument.

### FE-SEM and chemical analysis by energy-dispersive spectroscopy (EDS)

Samples of all the file systems were submitted for the FE-SEM and EDS testing. The EDS technique detects X-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analyzed volume. The polished specimens were sputter coated with a thin layer of conductive platinum in a sputter coating unit.

NCF values were statistically analyzed by one-way ANOVA with SPSS Statistics for Windows, Version 22.0 (IBM, Armonk, NY, USA). The significance level was set at 0.05.

## Results

### Cyclic fatigue result

The cyclic fatigue is checked by the NCF. The NCF for each instrument was calculated by multiplying the TTF to the applied rpm. Results were compared using one-way ANOVA. Statistically significant differences were detected in NCF values of the systems as follows: Hyflex EDM > PTG > Hyflex CM ($P < 0.05$). No significant difference was found between Hyflex EDM and NeoLix NeoNiti ($P > 0.05$).

### The mean results of cyclic fatigue resistance test

- File 1: Hyflex CM; 2.41 min: NCF = *723*
- File 2: PTG; 2.12 min: NCF = 636
- File 3: Hyflex EDM; 5.57 min: NCF = 1671
- File 4: NeoNiti NeoLix; 5.54 min: NCF = 1662.

### EDX analysis of NiTi composition

The surface texture of all the contemporary NiTi rotary instruments were observed at high resolution by FE-SEM, as shown in Figure 1, which showed rough surface in case of both EDM based files (Hyflex EDM and Neolix NiTi) due to the sparks generated during the electric discharge machining manufacturing process, whereas smoother surfaces in case of PTG and Hyflex CM due to the cutting the NiTi wire blanks during manufacturing process.

Percentage changes in the compositions in instruments were analyzed using EDX (Figure 2) which are shown in Table 1 demonstrating the changes in the overall compositions of different brands of unused NiTi instruments as detected using EDX analysis which show a decrease in the Ni percentage in case of all types of contemporary files systems as compared to its traditional 55 at %.

**Table 1: Quantitative results of elemental analysis with atomic weight percentage**

| S. No. | Name of the NiTi rotary instrument | Element | Weight% | Atomic% |
|---|---|---|---|---|
| 1. | Hyflex CM | Na K | 0.25 | 0.26 |
| | | Mg K | 0.15 | 0.14 |
| | | Ti K | 46.13 | 46.79 |
| | | Ni K | 51.95 | 46.87 |
| | | Si K | 0.21 | 0.18 |
| 2. | ProTaper Gold | C K | 7.35 | 3.32 |
| | | O K | 8.63 | 6.76 |
| | | Ti K | 43.94 | 42.37 |
| | | Ni K | 50.08 | 51.55 |
| 3. | Hyflex EDM | Si K | 14.15 | 31.95 |
| | | Ca K | 20.50 | 34.74 |
| | | Ti K | 48.35 | 53.43 |
| | | Ni K | 51.65 | 46.57 |
| 4. | Neolix:- NeoNiti | C K | 7.35 | 19.32 |
| | | O K | 18.63 | 36.76 |
| | | Ti K | 46.02 | 51.10 |
| | | Ni K | 53.98 | 48.90 |

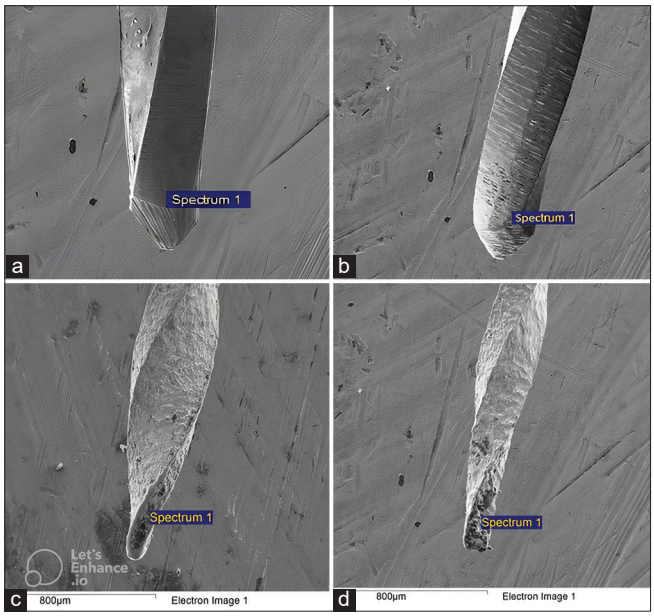


**Figure 1:** Field emission scanning electron microscopy (FE-SEM) images (a) Hyflex CM (25/0.06) and (b) ProTaper Gold (F2 - 25/0.08) both these files show smoother surfaces as compared to (c) Hyflex EDM (OneFile - 25/0.08) and (d) Neolix NeoNiTi (A1 - 25/0.06), both the EDM technology manufactured files have a similar rough surface is seen in FE-SEM.

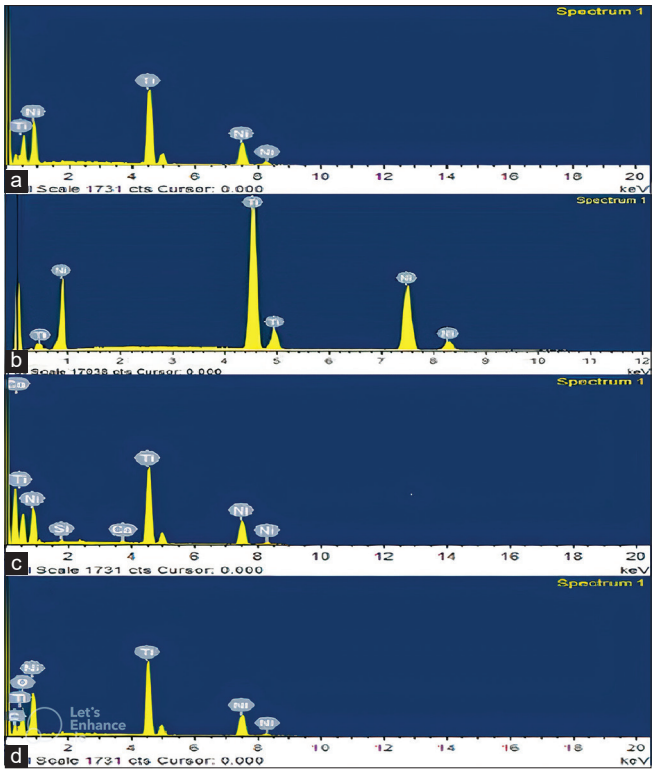


**Figure 2:** The EDS microanalysis of (a) Hyflex CM, (b) ProTaper Gold, (c) Hyflex EDM and (d) Neolix NeoNiTi indicates that all the tested instruments were composed of almost the similar equiatomic NiTi alloy. Si and Ca are seen in the composition. Trace elements of Sodium, Magnisum, and Silica can be observed.

## Discussion

Improvement in the manufacturing process of NiTi files is mainly due to the thermomechanical processing procedures. Despite these advances in metallurgy, still there are chances of failure/fracture of the instrument. The possibility of instrument separation is still a major concern during the clinical use of NiTi files.[5] Many variables contribute to file separation, but the two main causes are cyclic fatigue and torsional stress.[6]

To increase the predictability and the effectiveness of the endodontic treatment, with fewer incidences of instrument failure, there is a need to search for the best metallurgical alloy which has least chances of fracture and also increase in the mechanical performance. EDM process creates a rough and hard surface that could improve the cutting efficiency of tools. SEM of NeoNiTi and HyFlex EDM files revealed a rough "spark-machined" surface. HyFlex EDM is produced with the well-known controlled memory (CM) wire but with a different fabrication process that uses the ability of electrical sparks to partially melt and evaporate small portion of material in a dielectric medium.[7]

For PTG post heat treatment is applied after the flutes of a file have been manufactured. The temperature used is in a range of 370–510°C for a variable period of time. Files exhibit two

stage specific transformation behavior and high Af temperature around 50°C similar to CM wire. These findings explain the higher flexibility of PTG instruments at room temperature.[8,9]

Surface treatments carried out on EDM files. The decrease in NiTi content detected in the EDS test may be limited to the instruments' surface. As the oxide layer of the tested instruments is composed of metal oxides: nickel oxide and metallic nickel. There are significant differences in the fatigue resistance of the files tested, probably due to the different surface and alloy features.[10] The results of the present study suggested that endodontic instruments manufactured with EDM are expected to have higher strength and wear resistance than similar instruments made of CM wire and gold heat treated wires because of its unique nano-crystalline martensitic microstructure. Metallurgical tests are fundamental for thoroughly understanding the mechanical behavior of NiTi rotary instruments, particularly when the aim of the study is to compare the performance of different instruments. The combined data of AF temperatures from DSC and the alloy composition from EDS are used to check a correlation with the mechanical performance of a NiTi rotary file by the fatigue resistance tests, to bring out detailed information which allows clinicians, researchers, and engineers to understand the chemophysical rationale behind certain mechanical behaviors of endodontic instruments, giving further information for developing knowledge in this field.

## Conclusion

Based on present data, the advances in material processing appear to offer substantial benefits to the efficacy, efficiency. All samples showed surface irregularities. Hyflex EDM instruments demonstrated the highest NCF values in all the tested instruments. Hyflex EDM achieved the highest resistance to cyclic fatigue among the tested files. Such a study done in-depth will give insight to all the Clinicians/Practitioners/Doctors on which type of rotary file system to be used in which case. Further, completions of the study of metallurgical and mechanical tests are required to reach to a better conclusion.

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